

# INTERNATIONAL Chemical Engineering and Process Industries

Vol. 32, No. 11

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NOVEMBER 1951

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Subscription rates: 1 year 4 dollars, 3 years 8 dollars, paid in advance

England: Leonard Hill Limited, 17 Stratford Place, London, W.1

U.S.A.: British Publications Inc., 150, East 35th St., New York, 16

## Topics of the Month

### Planning to avoid shortages

ONE of the good effects of the sulphur shortage in Britain has been to focus the attention of the chemical industry upon the whole problem of raw material shortages. There is now a determination throughout the industry to do everything possible to avoid another humiliating crisis such as was precipitated by the American decision to cut brimstone exports. The Association of British Chemical Manufacturers has set up a special Raw Materials Shortages Committee to investigate the probable production and consumption over the next five years of products likely to be scarce. It is the sincere hope of the chairman of the A.B.C.M., in his annual speech, that 'our efforts will enable us to foresee, and by appropriate action avoid, situations like that created by the sudden reduction in supplies of American sulphur.' That, indeed, is our hope and the hope of all concerned with the British chemical industry.

Just how serious is the whole problem of raw materials shortages is illustrated by one case quoted in the A.B.C.M. annual report. This is dyestuffs, one of the premier sections of the British chemical industry. During the first half of this year the dyestuffs industry has been afflicted by scarcities of sulphur, sulphuric acid, naphthalene, bromine and

toluene, with the result that it has been necessary to increase imports of dyestuffs and intermediates. It is discouraging to have to resort to imports of finished goods through inability to procure the raw materials which would enable the dyestuffs industry to operate fully both to fulfil home demand and to sell abroad. It is with situations like this in mind that we welcome the efforts of the A.B.C.M.'s new committee and wish it well in its important task.

### Bring in the experts

THE Chemical Committee of the Organisation for European Economic Co-ordination is engaged on a big task. It is no less than an attempt to co-ordinate the plans of all Western European countries for the expansion of their chemical industries in order to avoid the installation of capacity in excess of all likely needs. Such a project for international rationalisation of an industry has seldom, if ever, been attempted before and no one knows whether it will succeed. What is important, however, is that every meeting of the committee should be able to draw upon expert advice. Most countries participating realise this and, as a matter of course, send representatives of their chemical trade associations along with their official government

representatives. In glaring contrast is the United Kingdom Government which, with rare exceptions, ignores the offer of help of the Association of British Chemical Manufacturers and, presumably, is content to send Whitehall planners only. This remarkable example of bureaucratic arrogance was given by Mr. C. G. Hayman, chairman, in his annual speech to the A.B.C.M. He was being purposely moderate when he stated that 'it is proposed to raise this matter with the authorities in order to try to bring about a more vigorous and realistic consideration of these problems.' We are able to put it more bluntly: throw out the 'planners' and call in the experts.

### British water pollution research

THE chief investigations made by the Water Pollution Research Board during 1950 were concerned with the development or improvement of methods of treating sewage and industrial wastes and with the assessment of the effects of polluting substances on natural waters. The report of the Director of the Board, Dr. B. A. Southgate, was recently published by H.M. Stationery Office and contains a brief account of this work.

Work has continued at Minworth on the treatment of settled sewage in percolating filters at rates very much higher than those normally used. This work is throwing some light on the mechanism of the process of biological filtration and this in itself would be sufficient reason for continuing it. In addition, it is very desirable to examine the possibilities of using this method on a large scale since, provided that an effluent of high quality is not essential, the volume of filtering medium required to remove a given amount of organic matter per day may be much smaller than that at present employed. A method of this sort might, for example, be of use in treating sewage, at a comparatively low cost, before discharge to an estuary or the sea.

Treatment to produce an effluent of unusually high quality has been studied in two series of experiments, both carried out at Luton. The first was on the mechanical filtration of sewage effluent, and this work has now been concluded; large-scale micro-strainers are already in operation there and an installation of sand filters designed from the results of work with pilot plant is scheduled to begin operation. The second investigation has been concerned with the further treatment of mechanically filtered sewage effluent at high rates in a percolating filter of special design.

Work is also being done by the Gas Research Board on the problem of disposing of gas liquors. In the past these liquors have sometimes caused serious difficulties at sewage-treatment works, particularly where the area served by the gas works is much bigger than that served by the sewage-treatment works to which the liquors are discharged. The Gas Research Board has arranged for a typical gas liquor to be separated into its various fractions at Leeds University and the effect of each of these fractions on the treatment of sewage is being examined, using small-scale plant erected at Stivichall. By making changes in operation at certain gas works it is possible to modify the composition of gas liquor, and the object of the work is to ascertain what modifications are most beneficial when the liquor is treated in admixture with sewage at a sewage-treatment works.

During the year under review, D. M. Newitt, Professor of Chemical Engineering at Imperial College, London, and immediate past-president of the Institution of Chemical Engineers, was appointed a member of the Water Pollution Research Board, of which H. W. Cremer is chairman.

### New chemical enterprises in Norway

NORSK Hydro, Norway's biggest chemical concern, plans to spend £12,500,000 to £15,000,000 on new plant to increase production of various chemicals. By installing improved electrolysis equipment Norsk Hydro's output of pure nitrogen will be increased by about 30,000 tons with no increase in the consumption of electricity. The increased production of nitrogen will enable the concern's production of nitrates, which last year totalled 1,000,000 tons, to be increased by another 200,000 tons. The new equipment will be installed at Norsk Hydro's factories at Glomfjord, Rjukan and Notodden. It will take a year to complete the conversion at Glomfjord. It has been decided to treble the production of concentrated fertiliser to 120,000 tons at Herøya and it is planned to double the production of urea. It is hoped to start construction of a factory to produce 30,000 tons of salt a year in the very near future. The production of magnesium oxide began this spring, and the first magnesium metal was turned out at Herøya in August. The raw materials are sea water and dolomite. Production of magnesium is provisionally 5,000 tons a year. It is hoped very soon to begin a considerable extension of Norsk Hydro's chlorine factory to increase production from 8,000 to 15,000 tons a year. Talks are proceeding with the Government with a view to building a phosphorus factory in north Norway.

This latter project will probably be part of the Norwegian Government's £10,000,000 development plan for north Norway which envisages the starting of enterprises to produce pyrites, cement, iron ore and textiles, besides a project for increasing hydroelectric generating capacity.

### New tin-nickel alloy

REMARKABLE corrosion resistance is claimed for a new tin-nickel alloy which may be electrodeposited. This property is so exceptional that the process, primarily designed to produce a new decorative finish, is now thought to provide a new constructional material for chemical plant.

The alloy has 65% tin and is said to be resistant at ordinary temperatures to alkalis, neutral solutions and nitric acid, and resistant to other acids at pH values greater than 1.2. It is also claimed to resist tarnishing in severely polluted atmospheres, being unaffected by SO<sub>2</sub> or H<sub>2</sub>S. It is also said to be unaffected by heating in dry air up to 320°C. at least. However, prolonged immersion in hot aqueous solutions, even distilled water, produces tarnish films.

There is some evidence that the alloy is covered by a protective film which, if removed, rapidly re-forms and, although slight corrosion in crevices where oxygen becomes exhausted has been observed, this effect seems likely to have little practical importance.

The evidence for these conclusions is given in 'Electrodeposited Tin-Nickel Alloy Coatings,' issued by the Tin Research Institute, which describes work done in the Institute's laboratories during the past two years. The first part relates to the method of the electrodeposition process and shows that the operating conditions are sufficiently flexible to ensure good sound deposits in actual workshop practice. The second part, dealing with the corrosion tests, puts forward an explanation of the remarkable corrosion resistance of the alloy which could not be predicted from a knowledge only of the corrosion resistance of tin and nickel separately. From this it is plain that, besides being a new permanent decorative plating, the alloy is an interesting chemical engineering material.

### Health hazards of welding

AN investigation into the possibility of ill health arising from the welding process has been carried out by the Factory Department of the Ministry of Labour and National Service and the results have been published in a book entitled 'The Health of Welders,' published by H.M. Stationery Office (3s., post free).

The investigation consisted of a survey of the various types of welding, followed by clinical examination of some 250 welders in different industries, supplemented in many cases by special examinations such as radiological examination of the chest and blood examinations. Literature on the subject, particularly that published during the past 15 years, has also been reviewed.

The main conclusions are that welders do not suffer from any specific disease that could be described as 'welders' disease,' nor does occupational dermatitis appear to be a frequent or serious cause of disability. Electric welders may suffer from 'arc eyes,' but this has no permanent effect on the vision. Electric welders also suffer to a greater extent than other workers from a slight superficial inflammation of the eyelids.

Among welders exposed to high concentrations of fumes slight irritation of the throat is not uncommon, but no serious effects on the throat or nose were observed. Symptoms, indicating a mild form of bronchial irritation, were higher than would be expected in a comparable non-welding group. Exposure to welding fumes does not, however, predispose to pulmonary tuberculosis. Stomach troubles are no higher in welders than in the general population, rheumatism is not excessive, there is no evidence of ill-effects on blood pressure or the nervous system and where there is good or moderately good ventilation there is little danger of gassing during welding.

Some general recommendations are made, but it is pointed out that, as the main risk of ill health among welders is due to fumes, the remedy is ventilation. The difficulty, however, is to decide when and where special provision should be made and, as there is such a wide variation in the different welding processes, it is considered that the problems of each firm will have to be looked at individually.

### Evaporation and freezing of milk

THE Canadian National Research Council at Ottawa has established a small laboratory to study certain dairy processes, such as low-temperature (about 60°F.) evaporation of milk. The older technique of high-temperature drying of liquid foods often produces slightly cooked or scalded flavours. Concentration by low-temperature evaporation is a relatively new approach to food preservation, but it is a technique which minimises these changes in flavour and, therefore, will find increasing application.

There is still much to be learned about optimum conditions of concentration, the extent of bacterial growth during processing and the storage life of the finished product. The possibility of freezing these concentrated milks has also to be considered. Whole milk can be hard frozen and stored at -10°F. for about four months. Concentrated milk can be frozen more economically because of its smaller bulk, but at present it only keeps for about two months at -10°F. After a few weeks in frozen storage the concentrated material cannot be reconstituted easily and the re-watered product tends to be chalky or even curdled. The object of the work at present being undertaken by the Council is to produce concentrated milk which can be preserved

in a frozen state for periods up to nine to ten months.

Milk concentrated to one-third its fresh volume by low-temperature evaporation is now on sale in several U.S. cities. In Canada, so far as is known, the only concentrated milk products on the market are those made by the older techniques. As conditions between the two countries do not vary widely, it is thought that this method of milk concentration could be satisfactorily introduced in Canada after further study.

### Bigger oil refinery for South Africa

WITH shrewd appreciation of the world's need for ever-increasing petroleum refining capacity, Standard-Vacuum Oil Co. of New York have decided to increase by more than 50% the size of their projected refinery at Durban, South Africa. The original plans, announced only last March (see these notes, April, p. 156), called for a 10,000 barrels/day refinery. Now, with ground-breaking of the site still many months away, the design has been altered to provide for a throughput of 15,600 barrels/day of crude. The cost will rise from \$12,500,000 to more than \$17,000,000.

Maximum flexibility in yields combined with compactness and relative ease of operation is provided for in the design of the new plant, now on the drawing boards. A combination-type unit, it will combine fluid catalytic cracking with crude distillation and thermal reforming functions. In addition to motor spirit, end products will include power and lighting kerosenes, automotive and industrial diesel fuels and light and heavy grades of fuel oil.

The constructional contract has been given to the Foster Wheeler Corporation. Several shipments of special equipment and materials have already been delivered. In spite of the increase in the size of the refinery, it is still scheduled to go on stream late in 1953 or early in 1954.

### Training a technologist, a chemical engineer's views

THE education and training of the technologist was discussed by Prof. F. H. Garner, O.B.E., in his Redwood lecture delivered to the Institute of Petroleum in London last month. Prof. Garner, who is Director of the Department of Chemical Engineering, University of Birmingham, said most scientists would agree that some knowledge of the humanities should be included in their training, but by no means all humanists consider that a knowledge of science should figure in theirs, although a man without some understanding of scientific methods and achievements suffers a serious impoverishment in his whole intellectual life.

In addition to some knowledge of the arts, the applied scientist, the technologist, needs special personal qualities of leadership which can only be gained by personal contact rather than through books.

Referring to the feeling current today that recent developments in science have endangered mankind's future and that this is in some way connected with the deficiencies in training and outlook of scientists, Prof. Garner said that the results of scientific discovery can be applied well or ill and it is the standard of intelligence and moral standards of the whole of the educated communities, not those of the scientists alone, which form the decisions. Moreover, until the last 50 years, had not the whole of the British educational system been inspired and conducted by those trained in the humanities? And the result of such training through hundreds of years is now said to be that the educated section of the community is unfitted to deal with today's problems.



Considering the broader aspects of training, Prof. Garner refuted the idea current in some quarters that applied sciences are out of place in a university and that modern engineering education consists of a series of snippets of subjects with no real grounding in any branch of science whatever. Some thought that engineers should be taught only applied mathematics, modern physics and recent advances in chemistry for three years and learn engineering in a graduate course. But surely the assumption that everything labelled 'mathematics,' 'physics' or 'chemistry' was basic or fundamental was wrong. It is a fallacy to assume that knowledge can be allocated into boxes, when in point of fact there is an immense borderline, such as for example physical chemistry, which is taught in chemistry departments but might equally well be taught in physics departments.

Discussing the factors necessary for a successful university training, he considered that the technologist requires a fuller series of lectures, tutorials and practical classes than is necessary for an arts student and these must be so directed that they lead to the development of the potentialities of the personalities of individual students.

The 'know-how' of industry can only be acquired in industry itself, but the teaching of why different techniques are used and the principles on which they are based can well be taught at the university. Contact with industry at home and abroad should be maintained through vacation courses.

Prof. Garner concluded by expressing the hope that graduate schools, so long an integral part of technological training in the United States, might soon be firmly established in Britain.

### **Sulphuric acid imports**

THE mishaps and misfortunes of the post-war years have played fantastic tricks with the normal economy of the United Kingdom. A few years ago the idea of importing sulphuric acid into a country ranking as one of the biggest acid producers in the world would have seemed just as absurd as the thought of importing coal. Yet both these things have happened. First, American coal has been unloaded at British ports and now, within the past few months, Continental sulphuric acid has become a British import.

Some details of the size of the acid-importing programme, made necessary by the cuts in supplies of American brimstone, are given in the *I.C.I. Magazine*. Imperial Chemical Industries Ltd. have taken the leading part in this trade. As long ago as the autumn of 1950 this company, paradoxically the biggest producers of sulphuric acid from indigenous raw materials (anhydrite), decided that immediate steps were necessary to import acid from the Continent. The biggest problem was transport. Clearly the usual means of transporting small quantities of acid—steel drums and carboys—were inadequate for the big operations envisaged. Sea-going tankers were the obvious means. Five tankers, with an aggregate capacity of 3,000 tons, normally used for carrying vegetable oils, were adapted for acid transport and by March they were ready. Up till August they had made 43 voyages between Spanish and French and British ports, bringing no less than 20,000 tons of acid. The greater part of these shipments has arrived at Runcorn, Cheshire, where I.C.I.'s General Chemicals Division have erected special equipment to deal safely and promptly with these hazardous cargoes. A big problem

arose from the absence of acid storage facilities at British ports. The only other way to discharge the ships quickly has been to have enough road or rail tankers assembled to receive the acid and take it to the factories where it is needed.

Although the quantity of acid imported is comparatively very small, perhaps only 3% of British output, it provides those marginal supplies of vital importance to some chemical manufacturers. However, there is no chance that it will ever compete with home-produced acid, since it costs 2½ to 3 times as much. Nevertheless, it is likely to be imported for most of next year.

I.C.I. and the other firms involved are to be congratulated on the way they have tackled the unusual and formidable task of importing acid, however much we deplore the circumstances which have made it necessary.

### **Rising chemical exports**

REARMAMENT, by monopolising a substantial part of the British engineering industry, is severely reducing this industry's important contribution to the export drive. Consequently, other industries must attempt to make good this loss. Obviously there is a limit to what can be done in this way—a foreign importer who has ordered £1,000 worth of oil engines is not going to meekly accept £1,000 worth of Turkish towels merely because it is easier for us to send the one instead of the other. However, other industries can help to mitigate export losses and one from which big things are expected is chemicals. Even though its first duty is to supply essential home needs, of which rearmament is by no means the least, the industry last year pushed exports to a value of £107,000,000, compared with £67,000,000 in 1947. In the first six months of 1951 chemical exports reached £66,000,000. On a volume basis this was nearly twice as much as the 1947 rate. What is more important is that exports to the U.S. are increasing; in 1950 they were valued at £5,860,000.

### **Clydach, 50 years old**

AS a result of Ludwig Mond's flash of genius while working with Carl Langer in a London laboratory, the Mond process for nickel was discovered and the Mond Nickel Co. began operations at Clydach just 50 years ago. The two men had produced nickel carbonyl more by accident than design. But there was no mistaking the patience and efficiency with which the chance observation was followed up. After semi-scale study in a Birmingham works, the Clydach plant took up the story, an army of workmen soon transforming the little Welsh village. At first a soft-roasted sulphide from Orford processing was brought as raw material from Canada. Later the process began with sinter or oxide containing a little sulphide and partially-reduced nickel. A series of successive volatilisers give an efficient extraction from residues containing decreasing proportions of nickel. In treating the residue of copper-nickel-precious metals by the Orford process, again use is made of a method born of a chance observation or 'lucky accident,' as it is called. For in the long struggle to separate nickel, or Old Nick's metal, which ever plagued old copper-miners, the accidental discovery of copper and nickel-rich material on a dump-heap in a works yard, with two distinct layers within the pot of 'metal,' gave the clue to an alternative to Mond's carbonyl process. The precious metal recovered at the Acton refinery complete a chapter on nickel history, on a metal so much in demand today.



# An Index of Mixing

## A STUDY OF THE UNIFORMITY OF MIXTURES OF POWDERED MATERIALS

By John A. Dukes, A.R.I.C.

The principles underlying a novel method of determining the heterogeneity of mixtures of powders are described in this paper. The idea was originally developed in the Chemical Research Department of Magnesium Elektron Ltd., and the results of the initial experimental investigations are outlined. An 'Index of Mixing' is proposed, giving a quantitative definition of the closeness of approach to the limit theoretically attainable in ordinary industrial plant.

THE industrial production of pure fused anhydrous magnesium chloride for electrolytic reduction to magnesium<sup>1</sup> is an example of a reaction occurring essentially between two solid phases and a gas. The process is represented most simply as:



followed by



Because the mean free path of the oxygen molecule is small under the industrial conditions of 800 to 1,000°C. and 1 atm. pressure, it is clear that the physical closeness of the magnesium oxide and carbon surfaces is of some importance in determining the rate of attainment of overall equilibrium. One may generalise that the degree of intermixture of the magnesium-bearing raw materials and the reducing agent must (within limits) be one of the factors controlling the rate of production of magnesium chloride.

Similar phenomena will occur in many other industrial processes. Indeed, it is often found that the intimacy of mixing of solid reactants is a rate-controlling factor in chemical reactions. It is, therefore, surprising that there is no generally accepted quantitative measure for homogeneity in mixed powdered materials. Attempts have been made by Lacey,<sup>2</sup> Beaudry<sup>3</sup> and others<sup>4</sup> to deal with this problem, but these have not yielded a solution readily applicable to the batchwise processes so commonly met with in chemical industry.

A paper by P. M. C. Lacey<sup>2</sup> describes work done concurrently with, but quite independently of, that dealt with in this note, and it sets out the nature of the problem with great clarity. An inconsistency is, however, apparent in the definition of the concept of an ideal mixture which denotes at one point a 'perfect' mixture of mathematical regularity and in another context a 'random' mixture of statistical perfection. We here distinguish between these two concepts and deal in detail only with the latter. Lacey's experimental techniques were most painstaking, yet they failed to yield a quantitative parameter describing the degree of perfection or randomness of any given mixture. He concluded, for example, that from his admittedly preliminary work 'it is probably impossible to express the state of

a mixture adequately by a single number' and he indicates that only graphical solutions are possible.

The treatment of the problem as discussed by Beaudry<sup>3</sup> is applicable in the main to mixing machines which take and deliver a more or less continual flow of powders. He uses, as we do, a set of analyses on small samples of the material as his initial data. He derives, from these data, a numerical parameter defining the efficiency of the blender. The results cannot, however, be applied to the common type of unit in which a batch of ingredients is placed, mixed and completely discharged before refilling. Of the machines dealt with by Beaudry the 'three possible types are: (1) Where one batch goes in at a time, inflow and outflow are simultaneous and the level in the blender is held constant; (2) where one batch goes in at a time, inflow and outflow are not simultaneous, outflow beginning at the end of inflow of a batch; and (3) when one batch goes in at a time, inflow is intermittent and outflow is continuous.' The type where the machine is emptied after each operation is clearly excluded.

Other investigators have dealt with the difficulty of the problem of batchwise mixing of solid powders or confined their attention to the mixing of liquids and gases.

The solution which is suggested below is distinctive in its approach to this problem and had already been confirmed as of value in some brief experiments early in 1943. The collaboration of Mr. C. J. Bushrod, Dr. Ivan Harris and Mr. J. T. Willock (all at that time members of research departments of Magnesium Elektron Ltd.) is acknowledged in the development of this solution and in the early analytical investigations.

### Empirical approaches to a solution

(1) **The mean coefficient of variation.** Several attempts had been made by the author and co-workers to arrive at a suitable parameter for the quantitative assessment and comparison of the uniformity of mixing of powdered materials using empirical approaches. None was successful.

It is natural, when considering the assessment of the uniformity of a batch of mixed powders, to start with a set of analyses on a number of small samples drawn from various parts of the batch.

In early work the mean coefficient of variation had been used as a measure of the variability between samples. The mean coefficient is an easily computed statistic given by

$$\frac{S(x - \bar{x}) \cdot 100\%}{n\bar{x}}$$

where  $(x - \bar{x})$  represents the numerical magnitude of the analytical deviation, irrespective of sign,  $n$  is the number of observations and  $\bar{x}$  is the arithmetical mean.

This mean coefficient was not in itself a good index of mixing for these two main reasons:

(a) The variation between samples will clearly depend upon the sample size, and

(b) The numerical magnitude will vary, for completely unmixed materials for example, with  $\bar{x}$ . Different results, therefore, would be obtained from a study of the coal in a coal-magnesia mixture and from the study of the magnesia in the same mixture. This is an inadmissible defect, since it is not possible to conceive of a mixture in which 'A' is well mixed with 'B' while 'B' is not well mixed with 'A'.

It was not possible to find a constant relationship, in the studies made, between the sample size and the mean coefficient, and the matter was not pursued further when the 'E.A.S.' index which is described below was discovered as a measure of homogeneity in random mixtures.

(2) **Perfect and imperfect mixtures.** It has already been indicated that there is a clear difference between a 'perfectly random' mixture of particles and a 'perfect' mixture. An approach to a treatment of the latter is outlined below, although it has not so far led to the derivation of any useful measure of homogeneity.

It is first necessary to define 'perfect' and 'imperfect' mixtures in terms of the independent variables and a definition of a 'perfect' mixture could be as follows:

A batch of  $B$  grammes of powder of  $p$  particles per g., containing an average proportion  $\bar{x}$  of a determinable constituent 'C' should comprise  $B\bar{x}p$  cells; each cell to consist of one particle of 'C' surrounded by  $1/\bar{x}$  particles of other material, 'not C'. The idea is easy to visualise except when  $1/\bar{x}$  is not an integer. Each cell would weigh  $(\bar{x} + 1)/p\bar{x}$  g.

A definition of an imperfect mixture, following naturally from the above, would

be of cells all of the same mass as the perfect, where some in the mixture are perfect, some 'all C' and the remainder 'not C.' If for this imperfect mixture the variance with respect to 'C' content, between cells, is  $m$ , it can be shown that the ratio of the numbers of cells of these three types is a set of functions of  $\bar{x}$  and  $m$  and that the sum of these ratios is unity. The cells would not be physically separable and would usually be too small for individual analyses. If in practice one takes  $N$  samples each containing  $y$  cells (so as to take  $Ny$  cells in all) the distribution is:

$Ny(1-m)$  cells are of the perfect type.  
 $Nym(1-\bar{x})$  " " " " "all C" "  
 $Nym\bar{x}$  " " " " "not C" "

But in any one sample there may be any or all of the above types of cells, dependent on the magnitude of  $y$ , and the possibilities of this statistical approach have not yet been fully explored.

The perfect mixture which has been defined above could only be attained by specialised equipment giving an ordered arrangement of particles. Such equipment is not inconceivable in theory but is well outside the range of plant for preparing reaction mixtures for chemical processes.

Any mixing or stirring of a perfect mixture would impair its perfection. Hence perfection of this kind is not attained by mixing and the best that an ordinary mixer can achieve is, fortunately, the random arrangement dealt with below.

#### 'Equivalent Agglomerate Size' (E.A.S.) of randomly mixed powders

The best mixture of two powders which an industrial machine is capable of producing is one showing a random distribution of particles. This concept is amenable to statistical interpretation. It also implies that in some localised parts of a sufficiently large batch of powder there will be relatively high concentrations of each of the separate ingredients—'by the laws of chance,' as it were. We will deal with a mixture of two ingredients, 'C' and 'not C,' and assume throughout that the particle sizes are equal and substantially uniform.

Then if a given mixture is not random with respect to separate particles it may still be random with respect to 'agglomerates' of particles of the separate powders. This is the fundamental idea upon which the solution of the problem is based.

It is not necessary that these 'agglomerates' should consist of masses of adherent particles, nor even that they need have any physical reality at all. What is to be assumed is only that certain analytical data be supposed to be representable by postulating the existence of such agglomerates.

If  $n$  small samples all of equal size are drawn from a relatively large batch of material containing a proportion  $\bar{x}$  of some determinable constituent 'C' and determinations of 'C' are carried out on each

of the  $n$  samples, the standard deviation of the individual determinations,  $x$ , is given by:

$$Ds = \sqrt{S(x - \bar{x})^2 / (n - 1)} \dots (1)$$

where  $Ds$  is the standard deviation and  $S(x - \bar{x})^2$  is the sum of the squares of the deviations of each  $x$  from the mean,  $\bar{x}$ .

If a sufficiently large number of samples is dealt with the mean  $\bar{x}$  is adequately represented by the arithmetical mean of all the determinations of  $x$ .

Now, when drawing a large number of samples, each of  $N$  individuals, from a randomly mixed population comprising a proportion  $p$  of one kind and  $q$  of another kind (where  $p + q = 1$ ), the standard deviation in the number of individuals of each kind in the samples is given by

$$Da = \sqrt{Npq}$$

Since there are  $N$  individuals in every sample, the standard deviation in the proportion of individuals in the samples may be written

$$Da/N = \frac{\sqrt{Npq}}{N} = \sqrt{\frac{pq}{N}} \dots (2)$$

If the terms in equations (1) and (2) are compared it will be seen that  $p$  is identical with  $\bar{x}$  and, since  $q = (1 - p)$ ,

$$pq = \bar{x} - \bar{x}^2 \dots (3)$$

Also, since  $Da/N$  corresponds with  $Ds$ , one may write

$$\sqrt{\frac{S(x - \bar{x})^2}{n - 1}} = \sqrt{\frac{pq}{N}} \dots (4)$$

and hence, from (3) above, squaring throughout and rearranging the terms,

$$\frac{1}{N} = \frac{S(x - \bar{x})^2}{(\bar{x} - \bar{x}^2) \cdot (n - 1)} \dots (5)$$

$N$  is the number of individuals contained in each sample where random distribution of individuals is postulated. Therefore  $1/N$  is the fraction of the sample with respect to which a random distribution may be assumed.

If in a mixture of two powders the particles are themselves randomly distributed, then  $N$  will be the number of particles in each sample. But if each sample contains  $g$  grammes and if the powders are such that there are  $P$  particles per g., each sample will contain  $Pg$  particles.

A fraction  $1/N$  of each sample of  $Pg$  particles may therefore be regarded as an 'agglomerate,' i.e. the agglomerate is  $Pg/N$  particles in number. From equation (5) we deduce, therefore,

No. of particles per agglomerate (the so-called Equivalent Agglomerate size, E.A.S.) =

$$\left( \frac{Pg}{\bar{x} - \bar{x}^2} \right) \left( \frac{S(x - \bar{x})^2}{n - 1} \right) \dots (6)$$

where  $P$  = no. of particles of powder per g.;  $g$  = no. of g. weight in each sample;  $n$  = no. of samples drawn and analysed;  $x$  = proportion of a determinable constituent 'C' in each sample;

and  $\bar{x}$  = mean proportion of 'C' in the mass of the powder.

The results of a typical investigation on an industrial mixing machine processing mixtures of coal and magnesium oxide averaging  $3.10^6$  particles per g. are summarised below:

| Mixing time (minutes) | Average sample weight (g.) | Equivalent Agglomerate size (particles) |
|-----------------------|----------------------------|---|
| $\frac{1}{2}$         | 0.978                      | 352,000                                 |
| 1                     | 0.8355                     | 17,970 (a)                              |
| 2                     | 1.450                      | 4,500                                   |
| 4                     | 1.285                      | 1,925                                   |
| 8                     | 1.226                      | 2,500 (b)                               |
| 16                    | 1.450                      | 1,040                                   |

#### Notes

(a) This set, consisting of 40 samples, could be divided into two equal groups, one of average weight 0.57 g. and the other of average weight 1.34 g. The corresponding E.A.S.s were 13,000 and 21,000 respectively. These results were near enough to the mean, tabulated above, to indicate that the E.A.S. might prove to be independent of sample size. Theoretically, the most valuable feature of the E.A.S. should be that it is independent of sample size, but this point still requires exhaustive experimental demonstration.

(b) It was at this 8-min. sample that the effect of experimental error in the analysis became evident. Samples over 1 g. in weight, and rough methods of determination of the coal in the coal-magnesia mixture, were clearly not precise enough for discrimination in so well mixed a powder.

#### General conclusions

For the purpose of comparing mixtures of various materials prepared by different machines, or mixed to varying degrees, the size of the samples and the methods of analysis must always be such that the observed deviations between individual samples is large compared with the experimental error. It will be equally evident that the samples must be drawn in a representative fashion and that they must be larger in size than the hypothetical agglomerates.

It will be appreciated that the agglomerates with which we deal in this treatment of the mixing index problem need have no real existence. Their supposed existence is only postulated in order to provide a convenient explanation for the non-uniformity which a series of analyses on random samples will in fact show. The agglomerate or collection of particles all of one type is therefore only a convenient fiction. It does, however, allow the desired index of mixing to be determined. It follows, from the treatment given above, that the smaller the hypothetical agglomerate the better the degree of mixing which it represents. In the limit, a mixture which is completely random in the statistical sense with respect to individual particles should have an equivalent agglom-

merate size (E.A.S.) of unity under ideal conditions. The limitations of experimental technique and analytical accuracy make this limit unattainable in practice. The mixture itself represents a perfection which is also unattainable in practice and the E.A.S. therefore remains a useful measure of mixing for practical purposes.

If the derivation of the E.A.S. is carefully considered it will be seen that it is possible to say of any given mixture :

'This material is not inferior from the point of view of heterogeneity (having regard to its "C" content) than a mixture of agglomerates each containing *E* particles of the separate constituents "C" and "not C" in a statistically random distribution throughout the material,'

where 'C' represents any one determinable constituent and *E* is the number (of particles) referred to as the Equivalent Agglomerate Size defined by equation (6) above.

The author thanks Magnesium Elektron Ltd. and the Ministry of Supply for permission to publish this work.

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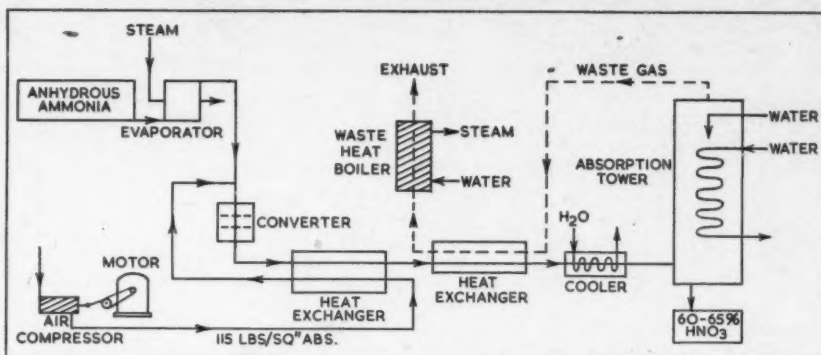
### Rubberised Paper

After spending three years and almost a million dollars on research the Du Pont Co. claim to have developed a new and economical way to add their synthetic rubber, *Neoprene*, to paper. Instead of coating or saturating the finished paper, as in older methods, the *Neoprene* is now added in latex form to the pulp just before it is made into paper. The result is a new range of low-cost special papers made directly on the paper machine which have high wet strength and chemical resistance.

These properties will give rise to superior paper products of many kinds, according to the company. One now under development is a floor mat for indoor or outdoor use. Woven of twisted rubberised paper, these mats have excellent abrasion and wear resistance; they can be washed with soap and water and hung up to dry without sagging or losing their shape. Double-wall insulated bags for cold bottles or cans of beer use *Neoprene*-treated paper so that the bag will not break when water condenses on the containers and soaks the paper.

In the industrial field *Neoprene*-treated paper has undergone successful field trials in such applications as gasket paper, multi-wall bag paper, wrapping paper, box board and industrial filter paper. Du Pont will not manufacture any of these papers, but will supply *Neoprene* latex and technical assistance to the paper industry.

## Energy Recovery in the Nitric Acid Pressure Oxidation Plant



Nitric acid plant with waste heat boiler.

IN the pressure oxidation process as used in the U.K., anhydrous ammonia and compressed air (at about 115 lb./sq.in.) are preheated, mixed together and passed over a platinum gauze, which results in the formation of nitrous oxide. The reaction is exothermic and the temperature of the gases is thereby raised to about 900°C. The rest of the process consists of cooling, condensing and absorbing the gases, whereby the nitrous oxide is further oxidised to nitrogen peroxide and absorbed in water to form nitric acid of about 60% concentration. The tail gases consist mainly of nitrogen and, since these are still at a pressure in the region of 100 lb./sq.in., they may be expanded through a gas turbine and the power thus made available employed for driving the compressors.

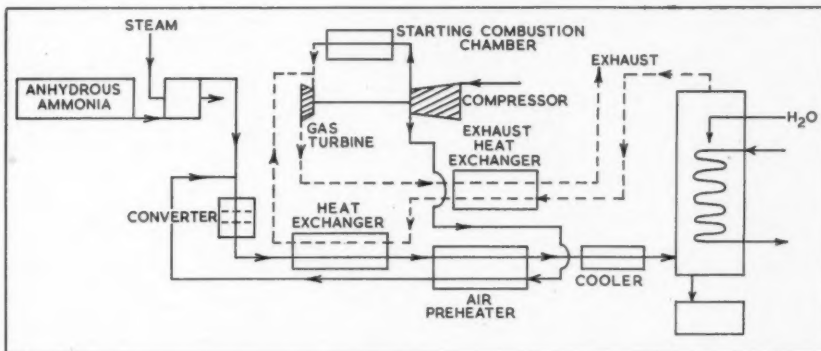
#### Modifications necessary

The modifications to the plant consist essentially of replacing the reciprocating air compressors and their electric motors (which aggregate some 1,100 h.p. in a typical plant) by a rotary compressor which is directly driven by the gas turbine. The cooler or waste heat boiler originally employed for cooling the gases is replaced by a heat exchanger and cooler of reduced size, the former reheating the exhaust gases from the absorber on their way to the turbine.

The idea of utilising the energy from the exhaust gases is not new and, in the past, reciprocating expanders have been employed with considerable success. However, these are not capable of utilising high-temperature gases and their efficiency is lower than that of a turbine. As a result, they are only capable of supplying a portion of the work required to operate the compressors and some external power supply is also necessary. Since the gas turbine is capable of supplying all the necessary power for driving the compressors, thus eliminating the need for an electric power supply, the saving in overall production cost should be very considerable and it is estimated to amount to at least 15%.

#### Starting up the plant

Another point of interest is the method used for starting up the plant. Since the turbine requires a supply of hot gas in order to develop the necessary power to drive the compressor, an 'auxiliary' combustion chamber is fitted for starting purposes. The 'converters' are by-passed on starting, whilst the air is led directly from the compressor to the turbine via the auxiliary combustion chamber (which may be arranged to burn kerosene or diesel oil). When full speed has been attained the flow is gradually switched over from the combustion chamber to the converters.



Nitric acid plant with gas turbine energy regeneration.



# PUMPING PRACTICE

## Pump selection, the N.P.S.H. problem, corrosion, gland sealing

By A. Flindle, M.I.Mech.E.

THE main types of pump used in the chemical industry are :

- (a) Positive-displacement direct-acting.
- (b) Rotary.
- (c) Centrifugal.

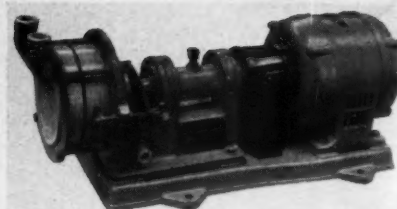
(a) In the case of the direct-acting plunger-type of pump this has two opposed plungers, and the pump is designed in such a way that a number of these units can be built together and driven by a single motor. With this arrangement parallel operation is easily achieved. A performance of, say, 200 gal./hr. at 600 lb./sq.in. pressure, would be typical for this kind of pump.

A variation of the direct-acting chemical pump is the glandless plunger pump, which was designed especially for handling

sulphuric acid, advantage being taken of the excellent lubricating properties of this acid by omitting the plunger stuffing box.

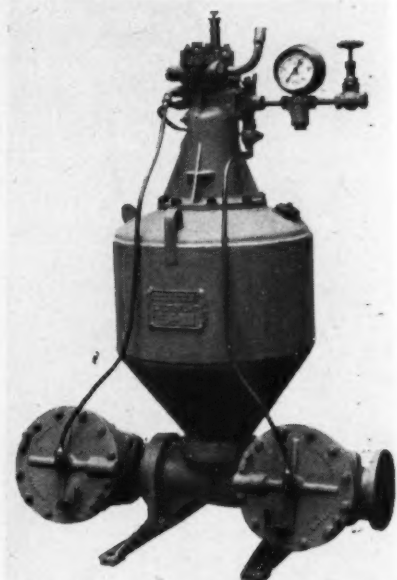
(b) The rotary pump gives positive displacement by the eccentric rotation of a helix rotor in a double-helix stator of rubber or synthetic rubber. This pump is exceptionally good for small outputs, and is being increasingly used.

(c) Centrifugal pumps are widely used in the chemical industry, and the most successful type has proved to be that with the simplest mechanical design. The pump must be of robust construction with internal parts easily replaceable ; glands, valves and other sources of leakage should be reduced to a minimum.



[Photo : L. A. Mitchell Ltd.]

Rotary-displacement self-priming acid pump. It is constructed in chemical stone-ware, so there is no risk of contamination and its use is not limited to any one type of liquor. It is suitable for handling all acids except hydrofluoric and is in service in many chemical works, proving useful for the emptying of carboys and boshes, and the transfer of liquor from one vessel to another. This model will deliver 780 gal./hr. when driven by a 1½-h.p. motor, against a total head from all causes of 20 ft., of which 10 ft. can be on the suction side.



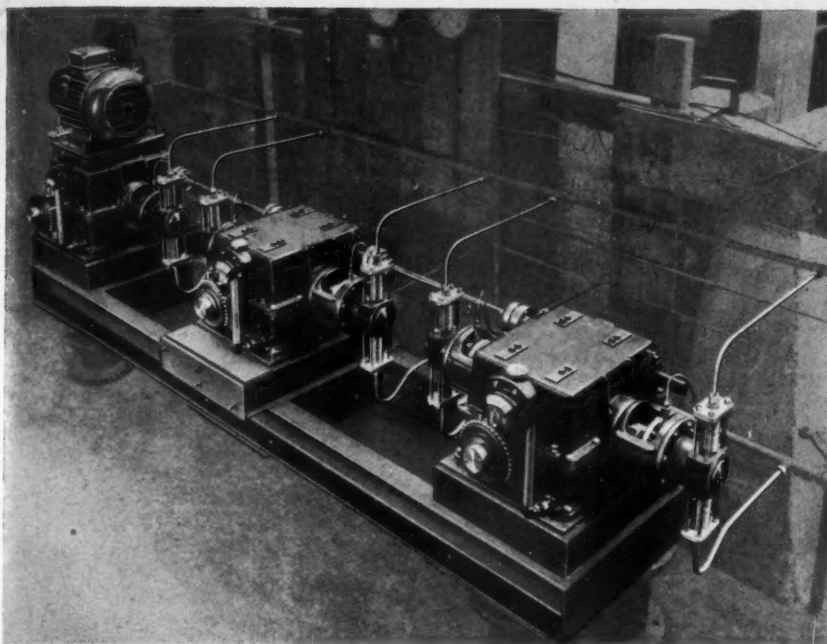
[Photo : Gresham & Craven Ltd.]

Compressed-air-operated 'Ejectopump.' It is purely pneumatic in action and consequently has no rotating or reciprocating parts. It operates automatically when connected to any compressed-air line and pumps the liquid to be handled in a series of regular suction and discharge strokes. Unlike most other compressed-air-operated pumps it can be arranged above the level of the liquid to be pumped as it is self-priming. The pump has no glands, bearings or revolving shafts and is claimed to require no lubrication. The pump casing is never completely emptied during normal working and no air passes into the delivery pipe. Therefore, it is impossible for aeration to take place and, as there is no churning action, the pump is particularly suitable for food products. As there is no vibration, special foundations are not required and the pump can be placed on any level surface. There is no fire risk when pumping inflammable liquids as there are no electrical connections to cause sparks. When supplied with compressed air at 30 to 50 lb./sq.in. the standard model will deal with discharge heads up to 50 ft. The normal maximum suction is 10 ft., but by fitting a special ejector, 20 ft. can be dealt with. The pump is produced in 1½, 2, 3, 4 and 6 in. sizes.



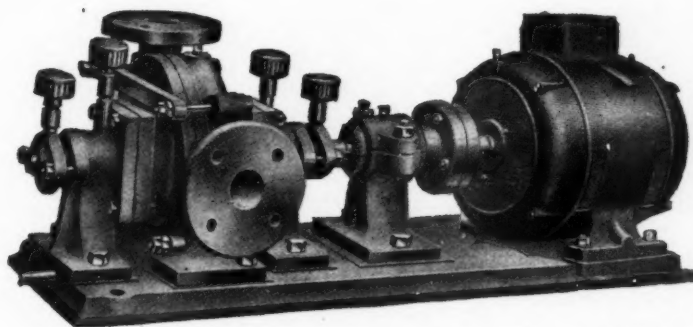
[Photo : Girdlestone Pumps Ltd.]

Twin-cylinder DMF duplex model of glandless diaphragm pump developed for processes where corrosive and abrasive liquids have to be pumped. It is of simple design, the driving shaft transmitting motion through a heavy connecting rod to a moulded diaphragm pulsating 375 times/min. The diaphragm is clamped to the pumping chamber containing the valves, which are readily accessible without removal of the diaphragm or pipe connection. A reinforced moulded-rubber diaphragm manufactured in various grades of rubber and synthetics to suit the liquid pumped is used. The pump body and pumping chamber is a one-piece casting of gunmetal, cast iron or ebonite-lined cast iron. For low-pressure applications, truncated moulded-rubber valves are used. For high-pressure applications, moulded-rubber disc valves are used, held in place by ebonite or stainless-steel screws on perforated ebonite or metal seats. Both types of valve are interchangeable. The liquid is completely sealed within the pump, stuffing boxes, rotary seals and such points subject to leakage and wear having been eliminated. It can be run dry indefinitely and there is no aeration and consequently no oxidation of delicate liquids. Among some of the chemicals handled by these pumps are ammonia, casein, plating solutions, penicillin, soaps, vitreous enamel and solutions containing jewellers' rouge, carborundum, diamond dust, ground glass and other abrasives.



[Photo : Candy Filter Co. Ltd.]

Three duplex, type G 'Minor' pumps each fitted with two special stainless-steel high-pressure heads incorporating oil seals. Instead of the standard stroke change handwheel, these pumps are arranged for remote variation of output by means of chains and sprockets from a control room on a higher floor. These metering pumps incorporate a new type duplicate suction and delivery valves in the stainless-steel high-pressure pump heads. These are flat valves, finely polished, usually made in Firth's F.D.P. quality stainless steel. The advantage of using this type of valve instead of ball valves is that it may be machined from a variety of materials, depending upon the chemical resistance required. In addition, it has been found that by careful design of the valve seat, pump efficiency has been still further improved and, with certain types, now reaches up to 96 to 98%. Valve design plays a very important part in precision pumps where constancy of output under varying conditions as well as a high degree of repetition accuracy is essential. Another new feature is the inclusion of an oil seal immediately beyond the packing; this solves the problem of handling liquids containing abrasive solids. The oil is maintained at a pressure somewhat higher than the working pressure and the assembly is so arranged that it not only reduces the wear on the packing very appreciably but also automatically compresses the packing. Candy metering pumps with pump heads constructed in materials other than stainless steel and suitable for handling concentrated acids, particularly corrosive liquids, are in general use in the chemical industry.



[Photo : Houghton's Metallic Co. Ltd.]

Centrifugal pump for sulphuric acid of all strengths up to 80%. The pump is arranged with a double-inlet shrouded impeller and bifurcated suction lead in such a manner as to give an evenly balanced impeller and to promote suction at the glands to avoid acid leakage. All parts in contact with the liquid are made of antimonial lead, with the exception of the pump shaft which is made of chromium-nickel-tungsten stainless steel. The casing and cover are held by wrought-iron clamps instead of nuts and bolts, for easy detachment. A new type of lubricator with automatic grease feed is fitted to the pump bearings. The unit is driven directly by an a.c. electric motor running at 1,440 r.p.m. The 1½-in. size pump will give an output of 33 gal./min. or roughly 2,000 gal./hr. of sulphuric acid to a head of 30 ft. At the moment the same type of pump is being made up to the 3-in. size, which will give 6,000 gal./hr. output at 50-ft. head.

The need for unchokeable pumps frequently arises in the chemical industry, and pumps having stoneware or vitreous linings are resistant to practically all acids with the exception of hydrofluoric. Stoneware or vitreous-lined pumps are also resistant to almost all alkaline products and have a reasonably high resistance to abrasion.

There appears to be many uses for the vertical glandless acid pump, which should be used far more extensively than it is at present. The omission of the gland and the possibility of installing the pump suspended in the acid itself are two very important features.

The various applications of centrifugal pumps to the chemical industry, particularly for handling acids of all kinds, have been admirably surveyed by Tetlow.<sup>1</sup>

The most persistent pumping problems in the chemical industry are probably the four listed below :

### 1. Pump selection for a particular duty

To make a satisfactory selection of pumping plant for a particular duty the conditions under which it must operate should be thoroughly investigated. In most cases it will be found that small rates of flow with high pressures will require a positive-displacement type of pump. This may probably be linked up with the possibility of small variations in rates of discharge over a wide range of viscosities.

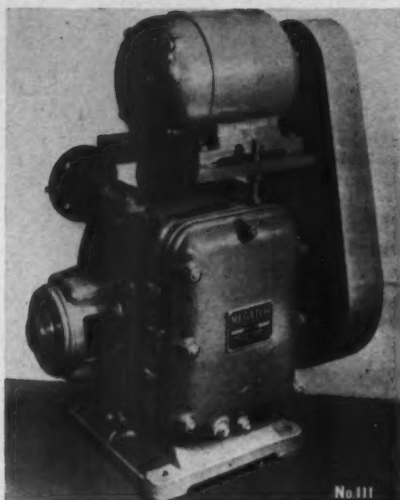
Positive-displacement pumps are essential for kinematic viscosities of 30 Stokes and upwards. Below these viscosities the application of centrifugal pumps is permissible.

One of the most important points in the selection of pumping plant is the fact that centrifugal pumps are capable of discharging at a uniformly steady pressure, and the rate of flow can be adjusted over a very wide range without any detrimental effect on the pump operation. It is most important that all questions associated with pump selection and layout be carefully analysed, to ensure safety and accessibility.<sup>1</sup>

### 2. The Nett Positive Suction Head problem

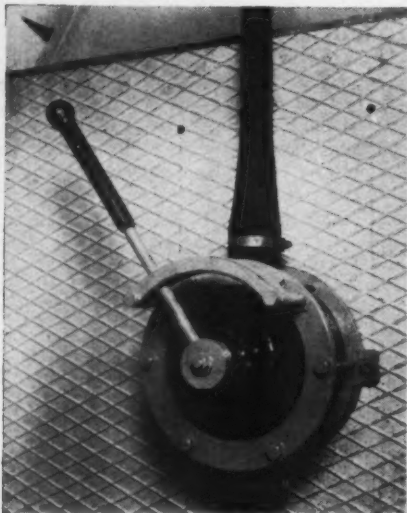
The release of gas or vapour from a liquid being pumped is a problem which must be considered in the design of chemical plant. It is even more important than the presence of solids in the liquid being pumped because, although solids can restrict the output, vapour locks can throttle the plant or prevent pumping altogether. Thus, pump design is controlled by pump suction requirements and, therefore, pump selection is controlled by plant design.

Gas entrainment, say 10% by volume, and the effect of insufficient N.P.S.H. has no great effect on the capacity of reciprocating or rotary pumps, but can cause severe reduction of capacity of centrifugal types.



[Photo: Megator Pumps & Compressors Ltd.]

A typical 'Megator' industrial pump with overhead-mounted motor. The pump gives the performance of a triple ram pump with the simplicity and compactness of a centrifugal. It is claimed to give unfailing self-priming, with a high-suction lift, and constant discharge over a wide range of delivery pressures. The low fluid velocity enables the pump to deal with thick and viscous liquids and an inherent characteristic of the design is that wear is taken up automatically. Maintenance is simple, and the pump can be opened up for inspection or cleaning by removing a cover and without disturbing the pipe connections. The wearing parts can be renewed in a matter of minutes without requiring any skilled fitting. Although this pump is not suitable for all conditions it is remarkably versatile. Liquids satisfactorily handled include barium chloride, chalk slurry, caustic soda, fruit juices, soda ash, syrup, olive oil, etc.



[Photo: Kastner Evaporator & Engineering Co. Ltd.]

Hand-operated semi-rotary hand pump designed to deal with very small quantities of liquids. This model is built in 'Keebush' and can be used safely with a wide range of corrosive chemicals, including hydrochloric acid. An output of 1 gal./min. against 10 ft. head can be obtained and the maximum head reached is about 20 ft.

[Photo: Gilbert Gilkes & Gordon Ltd.]

Self-priming pump (No. 250 GGG) fitted with clutch pulley. This design is manufactured in sizes from 1½ in. to 3 in. pipe connections. In the chemical industry this pump can be used for handling hot or corrosive liquids and in the petroleum industry as a suction lift for petrol or other volatile liquids up to 20 ft. without fear of vapour locking. It also makes an ideal float-controlled sump pump and can be used in breweries, laundries and dry cleaners for water supply, brine condensate, soap solutions and inflammable liquids. The pump has no metallic contacts, thus ensuring long life. The suction lifts up to 28 ft. of water and it delivers a non-pulsating flow discharging against a high head at low speeds. It can be constructed in phosphor bronze, aluminium bronze, stainless steel or other metals to suit most liquids.



If the N.P.S.H. is marginal, the reciprocating and rotary pumps are liable to suffer severe reduction in capacity, whereas in the case of centrifugal pumps these may only be moderately affected. N.P.S.H. difficulties are becoming more frequent, but everything has been and is being done to simplify the problem. Recommended charts showing nett positive suction heads should be reviewed in all cases of pump selection to suit specified duties.

There are various solutions to the N.P.S.H. problem and some are given in considerable detail by Irving Taylor.<sup>2</sup>

### 3. Corrosion and associated troubles

Corrosion is a major problem when selecting pumps for the chemical industry. However, different pump makers have made a large number of laboratory tests over long periods, with the result that today a wide range of materials is available for the different duties which may be encountered.

Acid pumps with stoneware, vitreous and rubber linings<sup>3</sup> are very successful, provided the lining is securely bonded to the metal body of the pump. To comment on corrosion in detail would take up considerable space. The experience of the various manufacturers of centrifugal pumps for the chemical industry must be relied upon for proper guidance.

A new pump has been developed by an American company which is claimed to resist wear and corrosion and to be highly successful when used by processors of milk, canned foods, carbonated beverages, liquors and a wide range of other products. It is a positive-displacement pump, made in five sizes, and precision-machined during manufacture. It is fitted with rotary seals, easily dismantled for quick and thorough daily cleaning. An important feature of

these seals, as noted in a later paragraph, is that the surfaces are highly polished and non-pitting to ensure efficient operation.<sup>4</sup>

Details have appeared of recommended materials for the pumping of acids, alkalis, oils, aqueous liquors, etc.<sup>5</sup> This information is very well compiled and offers considerable guidance in selection problems. In addition, a useful corrosion chart has been compiled by Tetlow.<sup>1</sup>

### 4. Gland sealing

The gland-sealing or stuffing-box problem must receive very serious consideration in order to ensure satisfactory operation under a wide range of conditions. For chemical work the characteristics of certain products must be considered and, although many acids may be more or less inert inside the pump, any leakage, however small, which may occur through the stuffing box can accelerate the rate of corrosion by contact of the acid with the moisture of the atmosphere.

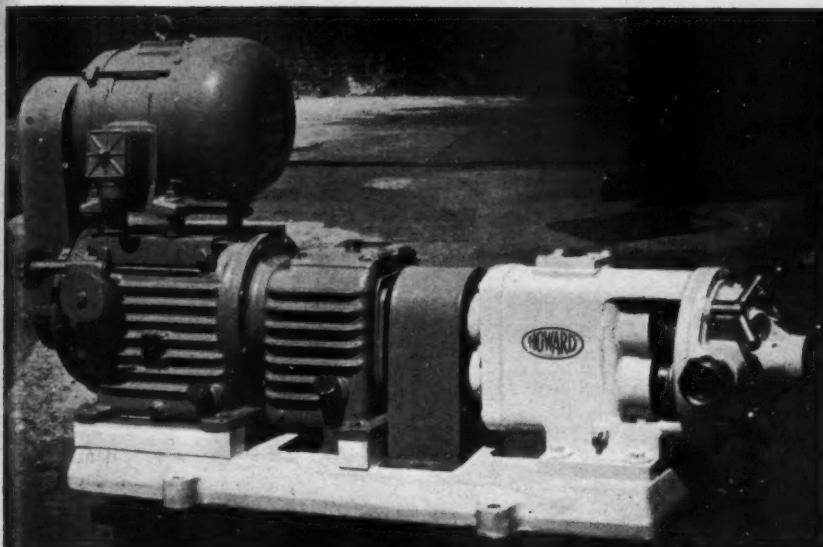
There are many and varied applications of gland sealing applied to pumps in the chemical industry, and considerable progress has been made with the use of mechanical shaft seals. One important feature of such seals is the circulation of the product round the seal faces in order to carry away any accumulated heat, and to ensure that wearing faces operate at permissible temperatures.

If lubrication of the seal faces is required, the lubricant is introduced directly between the surfaces in contact, and usually only a very small amount of lubricant is necessary. For this reason the mechanical seal finds many applications in the chemical industry where slight contamination of the product with lubricant can be permitted.

A number of interesting articles and

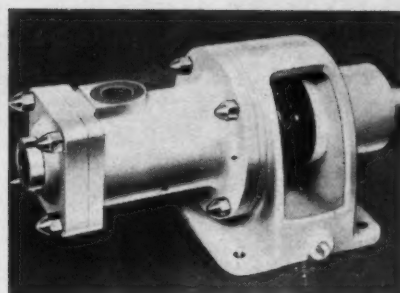
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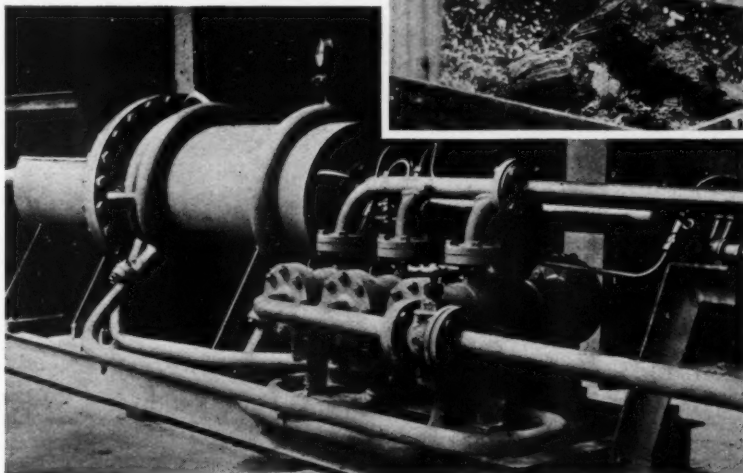
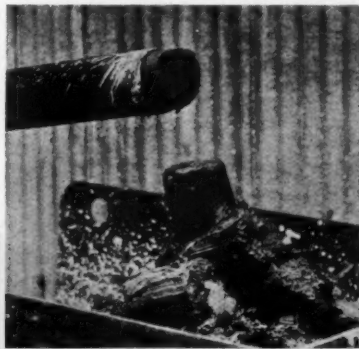
[Photo : Howard Pneumatic Engineering Co. Ltd.]

Rotary pump manufactured in Firth's F.M.B. quality stainless steel and driven through a Carter variable-speed unit. This model (size 4MD, type M) has been designed to meet the needs of the food and chemical industries. While specially suitable for pumping thick fluids, it will operate at electric-motor speeds when pumping water and other thin liquids and deliver them again against considerable pressures without wear. The pump has two rotors which rotate in opposite directions to each other, transferring the liquid from the pressure to the suction side. The rotors do not make contact with one another but always have a fine clearance between them when filled with liquid, forming a fluid seal and thus preventing excessive slip. For ease of cleaning the pump is constructed with a pump body overhung from the bearing housing, which makes all the working parts of the pump readily accessible and reduces the numbers of glands from four to two. These pumps are supplied in seven sizes, giving a range of duties from 150 to 20,000 gal./hr. for heads up to 150 ft. For a number of years they have been used for extracting milk and concentrates from vacuum pans operating under vacuum up to 29.5 in. Hg. They have also been supplied for pumping photographic emulsions, nitric acid, sulphuric acid, resins, acid sludge, caustic, etc.



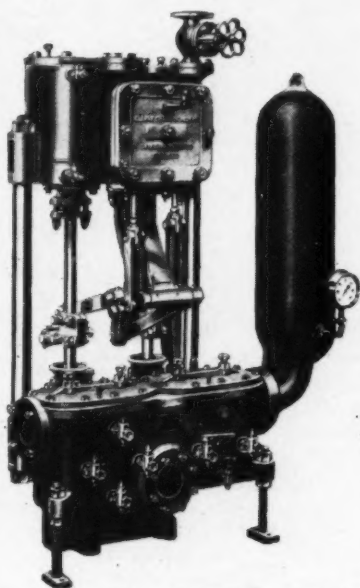
[Photo : Mono Pumps Ltd.]

In this pump (type IBD15) all metal parts in contact with the liquid being handled are of machine-finished stainless steel. The stator and flexible coupling can be of natural rubber or of synthetic rubber. By removing eight nuts the pump can easily be cleaned. The pump was primarily constructed for the batch production of cosmetics and pharmaceuticals, but its uses have been considerably extended for laboratory purposes, pilot plants and for sampling liquids. At a speed of 1,450 r.p.m. it gives a nominal capacity of 126 gal./hr. and the maximum pressure is 30 lb./sq.in. It is self-priming and will handle free-flowing or viscous fluids.



[Photo : Henry Balfour & Co. Ltd.]

This Meader pump is capable of handling thick sludge and semi-solids of such low moisture content that the angle of repose of the material is of the order of 90°. This feature is shown in the small photograph which depicts the discharge of thick clay-like process residue (bauxite of 30 to 40% water content) from the end of a 200-ft. pipeline which, in this case, actually contains five right-angled short bends. The pump is operated by hydraulic power obtained from a multi-stage centrifugal pump. The operating medium could also be either steam or compressed air, or by a direct mechanical drive. Among the advantages claimed is extreme flexibility. The speed, and therefore the output, is capable of infinite variation between zero and maximum merely by adjustment of the pressure fluid inlet valve. Another advantage is that the total quantity of material which has been pumped over a period can be determined by means of a simple counter with the disc. This feature only applies to those materials which will flow into the chamber freely enough to ensure that the cutting cylinder is completely filled at each stroke. Tests of the pump have demonstrated its suitability for handling thick and semi-solid substances such as various muds, clays, residues from many chemical processes, thickened sewage, cement slurries, unset concrete, ceramic compositions, pastes, etc.



[Photo : Clarke, Chapman & Co. Ltd.]

Vertical duplex soda and dilute acid pump. The unit is sturdily built, the liquid cylinder and fittings being supplied in non-corrosive material to suit the various duties involved and guaranteed to withstand acid and alkaline attack. It is designed especially for dealing with sugar, syrup, soda, dilute acids and similar products and is extensively used in the chemical and allied industries.

papers have been written on this most important subject as applied to the chemical industry, and all these indicate very clearly the increasing application of mechanical seals and the ingenious methods devised to overcome the many difficulties encountered.<sup>6, 7, 8, 9</sup>

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- <sup>9</sup>M. J. Dabney and W. W. Holt, Jun. : *Oil & Gas J.*, January 4, 1951, p. 57.



(Photo : Pulsometer Engineering Co. Ltd.)

A 12-in. high-vacuum chemical pump. It comprises a cylinder in which is mounted an eccentric rotor provided with two spring loaded blades. The rotor is set in contact with the cylinder at the top between the inlet and outlet branches. When the rotor revolves the blades form cells which increase from a minimum volume when passing the suction to a maximum volume at the end of half a revolution; they then decrease again to a minimum when passing the delivery port. This action compresses the medium to be evacuated and discharges it to atmosphere through the delivery valves. The cylinder, rotor and end covers are made from close-grained cast iron and the rotor blades are of steel. All springs are made from stainless steel. The rotor is carried on heavy-duty roller bearings. For certain classes of chemical work, where it is not possible to completely trap and retain all corrosive and condensable vapours generated in the plant, this pump is supplied with protected-type rotor bearings and the only working parts in contact with the gases are the rotor, blades and cylinder bore. It is also usually provided with drip-feed-type lubrication in which clean fresh oil is fed at a drip rate to the cylinder and after passing through the pump is allowed to overflow on the delivery side for collection, where the degree of contamination taking place in the oil can be checked and the oil cleaned before using again. This design of pump is also available in 6/20 in. and 8/50 in. sizes.

## Fractional Distillation

A NEW edition of a well-known textbook is always interesting. This is particularly so in the case of the fourth edition of 'Elements of Fractional Distillation,' for although it is only 12 years since the third edition appeared, in that interval theoretical knowledge of distillation has increased more rapidly than at any previous time and the earlier edition had become considerably dated. The greatly increased bulk of available material has forced the author to be extremely selective, but even so the volume has increased from 267 to 492 pages. Many of the older sections have been eliminated or abridged and more pages than those represented by the difference in size of the two editions have been given over to new topics. The preparation of the fourth edition has been done by only one of the former authors and this may account for the considerable change in emphasis throughout the book, a change which has produced a book catering for a different public and to which a title implying elementary treatment is no longer appropriate. The author is quite conscious of this and in a preface states that the book has been modified to fall 'more closely into line with the graduate instruction at Massachusetts Institute of Technology.'

Industrial practice in the distillation of ammoniacal liquor, methanol, ethanol, benzolised wash oil and crude petroleum, each of which was allotted one chapter in the third edition, have, in the fourth, rightly been eliminated as major subjects of discussion, although certain aspects of these processes appear throughout the book as illustrations of more general themes. Much of the more elementary physical chemistry of the earlier edition has also been discarded.

Apart from some discussion on thermal economy in distillation those chapters on the rectification of binary, multicomponent and complex mixtures remain substantially the same as in the preceding edition, the author minimising the importance of developments in this field as showing little advantage over long-established methods of calculation. Rather disappointing is the casual dismissal of the powerful Underwood equations for the determination of plate requirements and minimum reflux ratios.

Among those sections which are quite new to the book are a brief one on the experimental determination of vapour-liquid equilibria and a very extensive one on the calculation of such equilibria, the latter contributing in large part to the increased size of the volume. Excellent but rather brief chapters on azeotropic, extractive and batch distillation are welcome additions which might advantageously be extended by the utilisation of the space now devoted to vacuum distillation. It is unfortunate that much of the work of Rose and his collaborators has appeared too late for inclusion in the book as this sheds on batch distillation some of the light which the author sees to be so urgently needed.

Column design and performance are discussed at much greater length than formerly and this goes some way towards counterbalancing the large increase in physical chemical topics earlier in the book. Unfortunately this corrective effect is not continued in the final chapter in which column auxiliaries are inadequately discussed.

The book represents a considerable, if uneven, advance over the earlier edition and will be correspondingly more valuable to the chemical engineer, but may be of reduced value to other readers in search of more general instruction.

J. S. FORSYTH, PH.D., A.R.I.C.

### The Leonard Hill Technical Group

Articles published in some of our associated journals in the Leonard Hill Technical Group this month include :

Ciba's New Factory at Pyewipe, Grimsby ; Anti-TB Drugs ; Progress Reports on Antiseptics and Disinfectants, Perfumery and Essential Oils and Economic Poisons—MANUFACTURING CHEMIST.

Oil Tanker Progress ; Oil Transport by Road Tanker ; Oilfield Transport and Lifting Equipment—PETROLEUM.

Review of Pigment Progress : Wet Ground Mica ; Phthalocyanine Pigments ; Titanium Pigments Research—PAINT MANUFACTURE.

The Use of Chemicals in Food ; Food Control in Great Britain ; America's New Citrus Juice Plant ; Refrigeration and Ancillary Equipment—FOOD MANUFACTURE.

Improvements in DDT Spraying ; Biological Control of Insect Pests of Crops ; The Cocoa Industry ; The Cocoa, Chocolate and Confectionery Manufacturers' Conference—WORLD CROPS.

Plant on Hire ; Harbour Construction at Ghent—MUCK SHIFTER.

Focussing Principles of High-Energy Particle Accelerators ; Fission Products in Industry, Part 1—ATOMICS.

Design in the Pottery Industry ; A Galaxy of Glass, Part 3—POTTERY AND GLASS. Soft and Stiff Finishes in Textiles—TEXTILE INDUSTRIES.

Plastics, a New Building Material—BUILDING DIGEST.

### 'I.C.E.' December

Articles will include : Size Reduction Review ; World Chemical Conference ; New British Lubricants Plant.

\*Elements of Fractional Distillation, by C. S. Robinson and E. R. Gilliland. Fourth edition. McGraw-Hill, New York, 1950, pp. 492, price 59s. 6d.



# International Heat Transfer Conference

The subject of heat transfer covers more than the design and operation of boilers, condensers, coolers, radiators, air heaters and similar equipment developed especially for the transfer of heat; problems arise in all heat engines and devices where heat is generated, either intentionally or incidentally. An International Heat Transfer Conference was recently held in London. It was organised jointly by the Institution of Mechanical Engineers and the American Society of Mechanical Engineers. About 100 papers were presented and discussed during the three-day meeting. The papers were divided into five sections dealing respectively with heat transfer with change of state; heat transfer between fluids and surfaces; conduction in solids and fluids; radiation, instrumentation, measuring techniques and analogies; and special problems. Nine papers of direct chemical engineering importance are summarised below.

## HEAT TRANSFER WITH CHANGE OF STATE

### Effect of vapour velocity on condensation inside tubes

INDUSTRIAL practice has indicated that often much higher coefficients of heat transfer are obtained when vapours are condensed inside tubes rather than outside. F. G. Carpenter, National Bureau of Standards, Washington, D.C., and A. P. Colburn, University of Delaware, described investigations carried out to determine the heat transfer rates obtained when condensing a pure vapour inside a vertical tube under varied conditions of vapour velocity, tube length and physical properties of the vapour being condensed.

Pure vapours of steam, methanol, ethanol, toluene and trichlorethylene were condensed inside a vertical tube 0.459 in. i.d. and 8 ft. long, with inlet vapour velocities up to 500 ft./sec. Temperatures of the cooling water were measured by thermocouples located in the jacket at 6-in. intervals. The vapour temperature was measured at any point along the tube axis by a travelling thermocouple. The data were calculated as average coefficients of heat transfer for the entire tube, and also as local values at various points along the tube.

The coefficients of heat transfer for the condensing vapour were much higher than would be predicted by the Nusselt equations, even the relation including the effect of vapour velocity on the viscous film thickness. It was hypothesized that the condensate layer becomes turbulent at much lower values of Reynolds number than in the absence of a vapour velocity, and that the main resistance is in a laminar sub-layer whose thickness can be calculated from generalised velocity distributions developed for one-component flow in pipes. The experimental data was found to give good support to this theoretical approach. The application to design was outlined.

For a correct design of condenser with vapours inside the tubes, an average value of heat transfer coefficient should be predicted at a number of points along the tubes and then a graphical solution used. For an approximate design of a condenser with vapour inside the tubes, an average

value of condensing film coefficient of heat transfer for the entire tube may be estimated by the equations given. The justification for this is that most of the condensation in this type of unit occurs under the conditions of the condensate layer having turbulence induced by vapour friction. The reasonably good agreement with the present data provides confidence when the conditions are not too different.

### Condensation of benzene in the presence of air

The design of a condenser or dehumidifier for removing a condensable constituent from a non-condensing gas is far more complicated than that of a condenser to handle a single pure vapour. It is also very much more uncertain, for the problem has received little attention, either theoretical or experimental. Julian C. Smith, Cornell University, New York, and Horace T. Robson, Union Paper & Bag Co., Savannah, Georgia, discussed studies undertaken to provide at least some experimental data for comparison with values predicted by existing design methods, and to compare the results obtained with upward and with downward flow of the vapour.

Superheated mixtures of benzene vapour and air at about atmospheric pressure were passed at various rates either upward or downward through a vertical single-tube condenser, counter-currently to the flow of cooling water. The condensed benzene was returned to the boiler. The rates of flow of the air, the condensed benzene and the cooling water were measured, as were the temperature of the condensate, the temperatures of the vapour-gas mixture entering and leaving the condenser, and the terminal temperatures of the cooling water.

The condenser was a vertical 12-ft. (366-cm.) length of No. 15 B.W.G. copper tubing, 1½ in. (3.49 cm.) in outside diameter and with a wall thickness of 0.072 in. (1.83 mm.). It was held concentrically inside a length of 1½-in. steel pipe, which had an inside diameter of 1.61 in. (4.09 cm.). A tee was screwed on each end of the steel pipe. In the run of the tees a rubber ring held the copper tube in place; through the branch the cooling water entered or left the unit.

The length of the cooled section of the tube was 10.23 ft. (312 cm.).

Conclusions of general validity could not be drawn from the very limited experimental data presented. However, with mixtures of air and a typical organic vapour, downward flow through vertical tubes was much to be preferred over upward flow. When the vapour flow became streamline, existing design methods were conservative when downward flow was used.

With upward flow at low vapour rates, and with downward flow and turbulent vapour flow throughout the exchanger, the design methods were accurate for the specific conditions used.

Future work on this problem should involve studies of very short condensers or condenser sections, so that point values may be established accurately.

### Heat transfer by spray cooling

There appears to be no published work on the measurement of heat transfer rates for a hot metal surface cooled by a water spray, although there are two important reasons for determining their relationship to the other variables of the system. Firstly spray cooling is used widely, and until such a relationship is established the design of a spray cooling system must be empirical, and probably inefficient. Secondly, it may be necessary to choose between water flowing tangentially over a cooled surface and a system of sprays. At this moment this can be done only by trial. E. T. Linacre, Imperial College of Science and Technology, London University, described the results obtained in the course of such a trial carried out on behalf of the British Shipbuilding Research Association.

The variables involved are the nature of the cooled surface and of the coolant, which is usually water, and the geometry of the jet arrangement. It seems justifiable to neglect the nature of the cooled surface, beyond stipulating that it be reasonably flat and smooth. A second simplification follows from the experimental results that the heat transfer for a jet is independent of the distance between the jet orifice and the vertical cooled surface so long as the jet remains coherent and only slightly deflected by gravity. As a result, it follows



that the jet arrangement can be characterised by a single dimension  $D$ , the equivalent diameter of the area cooled by each jet.

The kinetic, heat transfer and thermal absorption effects of the coolant are represented respectively by the Reynolds, Nusselt and Prandtl numbers. Another term has to be added, the ratio of  $d/D$ , where  $d$  is the jet orifice diameter. Hence  $Nu = A(Re)(Pr)^m(d/D)^n$  where  $A$ ,  $l$ ,  $m$  and  $n$  are constants to be determined by experiment. If it is assumed that the temperature of the cooling water changes very little, the Prandtl factor may be omitted. When the cooling water is heated by the cooled surface to its boiling point, the constants of the equation will be quite different and it may be necessary to add terms allowing for the buoyancy of the vapour and the temperature of the coolant.

In the few experiments which have been carried out, the effect of a spray cooling a vertical copper surface have been studied. The method is suitable for measuring heat transfer both when the coolant is only slightly warmed and when it is raised to the boiling point. The basis of the procedure is the measurement of the thermal gradient along a copper rod (the 'probe') between the cooled copper surface and another parallel copper plate heated by an oxyacetylene flame. The temperatures of points along the probe are measured with thermocouples, and the probe is enclosed in a cylinder acting as guard ring. The temperature measurements are plotted on a graph and the line joining them extrapolated to give the temperature of the cooled surface.

The heat transfer coefficient (defined in terms of calories/cm.<sup>2</sup>/sec./°C.) was found to be independent of the surface temperature, which justifies the method of derivation. The result also showed that when the cooled surface is below 100°C. there is a layer across which heat conduction is Newtonian. Cleaning the metal surface had no effect, so that heat transfer was not being influenced by an appreciable oxide layer. Another set of experiments showed that the localisation of effective cooling increases when the water flow rate is increased.

#### Performance factors in the design of chemical heaters and coolers

Several hundred critical tests have been carried out on the many heaters and coolers in operation throughout the Billingham Division of Imperial Chemical Industries Ltd., and results given for liquids and gases at pressures from 1 to 360 atm. abs. R. A. Smith reported that in each case the ratio of the theoretical to the achieved overall heat transfer coefficient was calculated, and was shown to vary from 1.1 to 4, according to the cleanness of the fluids and the duty required. Difficulties were often encountered when testing equipment that was part of a chemical plant: flow

rates were not steady, instrumentation was inadequate, tests had to be carried out at a time to suit plant operation, and no changes could be made in the flow rates or temperature conditions. In particular, tests could rarely be carried out on a new and clean heat exchanger, but it was apparent from the results that the deposition of dirt on the tubes was the main reason for performance being poorer than that predicted from simple laboratory experiments in which clean tubes were used. The values obtained for the conductance of the dirt film could be correlated roughly with the amount of the deposit, and tests of the rate of build-up of dirt on different surfaces, from cooling water flowing at different velocities, were reported for an experimental cooler.

Discussion of the results in relation to the design of heat exchangers showed three factors which must be considered which may cause the observed overall heat transfer coefficient to be less than predicted. These are: (1) Stationary pockets of fluid may exist outside the tubes, owing to the effect of the baffles or to the fact that the branches cannot be immediately opposite to the ends of the tubes. (2) Perfect distribution of the fluid between the tubes of a multi-tubular heat exchanger is an ideal never attained in practice. The reduction in performance due to maldistribution is greatest when the outlet temperature of the maldistributed fluid approaches closely to the inlet temperature of the other fluid. It can be shown by mathematical analysis that, in all tests reported by the author, the effects of the likely amounts of maldistribution are negligible, except in the interchanger of one particular case cited. (3) Dirt is the most important factor and can be predicted approximately from test results. Thus the more reliable method of allowing for dirt is to estimate the dirt coefficient, rather than the conductance factor. An interesting example of this is afforded by the coolers of the five-stage compressors. In the first-stage coolers, the gas coefficient is the controlling factor and therefore low conductance factors are obtained; in the later-stage coolers, the gas coefficient is higher and so the dirt coefficient is the controlling factor, and higher conductance factors are obtained. In all stages the dirt coefficients are approximately the same.

The rate at which dirt is deposited on the inside of tubes from various cooling waters is now being studied. Results so far obtained with chlorinated River Tees water show that dirt is built up at a uniform rate (that is, the reciprocal of the dirt coefficient increases linearly with time), and that this rate is independent of the metal of which the tubes are made, provided that the surface has not been roughened by corrosion. The water contained 20 to 70 g./m.<sup>3</sup> solids in suspension and the deposits of dirt were made up of 40% organic materials and 48% iron oxide, the remainder being clay and river dirt.

The following results show how at higher water velocities the amount of dirt deposited is reduced:

| Velocity of cooling water, ft./sec. . . . .   | 2  | 4   | 6   |
|---|----|-----|-----|
| Dirt coefficient after three months, C.H.U./ft. <sup>2</sup> /hr. /°C. (or B.Th.U./ft. <sup>2</sup> /hr./°F.) . . . . . | 90 | 120 | 170 |

These figures are mean values for tubes of five different metals. The tubes were 21 ft. long and of  $\frac{1}{2}$  in. bore, and were heated on the outside by clean water entering at 90°C. (194°F.). The volume of dirt deposited in each tube was measured and the thickness of the deposit calculated. From these and the observed dirt coefficients it was calculated that the thermal conductivity of the deposit was 3 to 4 C.H.U./ft.<sup>2</sup>/hr./°C./in. This confirmed that the conductivity of a loose deposit, such as was obtained, is approximately equal to that of water which at 20°C. (68°F.) is 4.1 C.H.U./ft.<sup>2</sup>/hr./°C./in. With harder deposits the conductivity is higher; for example, in serial number 18, the thickness of the deposit was about  $\frac{1}{2}$  in., giving a thermal conductivity of 7 C.H.U./ft.<sup>2</sup>/hr./°C./in.

#### HEAT TRANSFER BETWEEN FLUIDS AND SURFACES

##### Shell side characteristics of shell and tube heat exchangers

A complete study of a commercial shell and tube heat exchanger was detailed by T. Tinker, Ross Heater & Manufacturing Co., U.S.A. This has pointed the way to design improvements that very substantially improve the heat transfer capacity and pressure loss characteristics of this type of heat exchanger. A recently designed tube bundle incorporates a number of these features as follows:

(1) The tube to baffle hole clearance is approximately 0.010 in. to reduce hole leakage to a minimum.

(2) The baffles are only 0.062 in. less than the seamless copper shell i.d. to keep baffle leakage small.

(3) The  $\frac{3}{8}$  in. tube o.d. permits a large amount of surface in a small package.

(4) The floating head closure is designed to permit the tube bundle to extend within 0.21 in. of the inside of the shell.

(5) All outer tubes are on a circle as close to the shell as possible to give a uniformly small by-pass clearance between tube nest and shell.

(6) The reduced leakage and by-pass areas indicated above result in the bulk of the shell side fluid flowing through the heat transfer surface, so that wide baffle spacing is sufficient for maintaining a high heat transfer coefficient with a minimum expenditure of pumping power.

Careful attention to design details as here illustrated can result in heat exchangers that are most economical of

construction materials, pumping power and space requirements.

### Heat transfer to flat surfaces

Heat transfer measurements made between air and a horizontal heated plate, where the air impinged vertically upon the plate from a multitude of holes, slots or nozzles, were described by S. J. Friedman and A. C. Mueller, E. I. du Pont de Nemours & Co. Inc., U.S.A. These tests were designed to simulate the conditions to be expected when heating or cooling continuous sheets of material passing underneath perforated plates; therefore, the lateral flow of air (parallel to the heated and perforated plates) was restricted to one or two directions by boxing in three or two of the sides of the rectangular test plates. The purpose of these tests was to determine the effect of hole size and spacing, distance between the perforated plates and the heated plate, and air velocity on the heat transfer rates from the plate and on the power consumed in forcing the air through the perforated plates and against the heated plate.

The results showed that the best type of air flow is from perforated plates which have a free area of 2 to 3% and are spaced about 4 to 6 holes diameters from the sheets. Some non-uniformity of heat transfer across a sheet is indicated, although it is small (around 10% on the average). Localised areas immediately under a jet may show larger deviations.

Attempts to compare the results with other studies on jet performance, on the basis of velocity decrease with distance from the jets, failed, due to insufficient range of experimental data and to the complexity of the flow patterns.

### RADIATION, INSTRUMENTATION AND MEASUREMENT TECHNIQUES

#### Heat transfer in a bed of fluidised solids

Heat transfer measurements in a fluidised bed of carborundum, coke, iron oxide ( $\text{Fe}_2\text{O}_3$ ), Devarda's alloy, and lead with air, various mixtures of nitrogen and hydrogen, argon, carbon dioxide, town gas, and raw methane as fluidising gases were described by Dr. C. van Heerden, P. Nobel and Dr. D. W. van Krevelen. A series of homogeneous sieve fractions as well as some mixtures of different sizes of coke and carborundum were investigated, the particle diameter varying from 50 to 800  $\mu$ .

All experiments were confined to 'dense phase fluidisation,' that is, at such a rate of flow that the expansion of the bed remains relatively small.

The very simple measuring apparatus consisted of a cylindrical brass cooler measuring 8.5 cm. in i.d. and 10 cm. in length, which was fixed between two glass cylinders of exactly the same i.d., the lower one being 10 cm. and the upper one 40 cm. in length. The heat transfer coefficient to the inner wall of the water-

cooled brass cylinder was measured. For this purpose just above the filter an electrical heating coil was provided, which develops a known amount of heat. The bed, which was filled with the solid powder to a few centimetres above the upper end of the cooler, will now adopt such a temperature that this heat can be removed by the cooler.

At a cooling-water temperature of 13 to 15°C. it proved to be possible always to regulate the heat production in the coil so that the bed adopted approximately room temperature. In this way heat losses to the surroundings were practically out of the question and, moreover, the amount of heat added by the passing gas and resulting from the difference of the temperature of the bed and the gas inlet was only a very small correction on the amount of heat developed by the coil. As a rather superfluous precaution the apparatus, except the cooler, was insulated by a coat of wadding.

The temperature of the bed was measured with a copper-constantan thermocouple, the bare junction of which was placed in the middle of the bed, halfway up the cooler.

From the inner surface of the cooler, the temperature difference between cooling water and bed and the heat developed by the coil, the overall heat transfer coefficient can be calculated. The amount of water (12 litres/min.) flowing through the annulus of the cooler (filled with glass beads to improve the heat transfer) was so large that the overall transfer coefficient could be identified with the heat transfer coefficient from the fluidised bed to the cooler wall.

From the results, it can be said that in the region of viscous flow ( $\text{Re} < 5$ ) a correlation on a basis of  $G/G_0$  is very satisfactory, while a correlation on a Reynolds number basis is possible only when the experiments for which  $G/G_0 < 2$  and  $> 20$  respectively are excluded, where  $G$  = mass velocity of gas based on empty tube,  $G_0$  = critical mass velocity at which fluidisation starts and  $G/G_0$  = reduced mass velocity.

Probably in the region  $\text{Re} > 5$  none of the two correlations will satisfy. Comparison of pressure loss measurements in a fixed bed with the velocity of free fall according to the Newton law, brings out that, in the region of complete turbulence ( $\text{Re} > 100$ ), the transition from dense to dilute phase fluidisation will occur already at a  $G/G_0$  value of 3. This means that in the region  $5 < \text{Re} < 100$  this transition will occur at a gradually decreasing value of  $G/G_0$ . It may be that in this region it is necessary to introduce the density of the fluidised bed.

### SPECIAL PROBLEMS

#### Heat transfer properties of mercury

Attention has recently been focused on the liquid metals as heat transfer agents for power-developing systems associated with atomic energy. Mercury has been used in

binary vapour cycles in the United States for some considerable time, and with a large measure of success after initial difficulties had been overcome. A survey of available literature on the subject of heat transfer to this liquid metal revealed some lack of knowledge of its properties under conditions of high heat flux (500 watts/cm.<sup>2</sup>), and work was initiated to extend the range of experimental data. It was described by D. English and T. Barrett, Atomic Energy Research Establishment, Harwell.

Generally, experimental apparatus for measuring the heat transfer properties of mercury involves the flow of mercury through a heated or cooled tube and, owing to its high heat transfer coefficient, the difficulty arises of separating the temperature difference owing to heat transfer from that owing to the thermal resistance of the tube wall.

The use of a conventional counterflow heat exchanger was rejected because of the difficulties of construction and of the separation of inner and outer coefficients.

A direct electrical heating method was eventually adopted, the heat being developed in the tube wall itself by means of heavy alternating current. By suitable proportioning, the heat generation in the mercury itself and the radiation and convection losses could be kept negligible and, by making the tube wall thin, temperature gradients within the tube wall could be kept to a minimum.

Throughout the series of experiments the maximum tube temperature was kept approximately constant at 200°C. (392°F.), a compromise between obtaining high heat fluxes and preventing surface oxidation of the copper. Experiments were performed ranging from a low Reynolds number of about 4,000 up to a maximum of some 45,000, the limits being set by instability and maximum flow obtainable respectively. The power input thus varied with the Reynolds number to maintain the arbitrary maximum tube temperature.

Measurements of mercury flow rate, electrical power, voltage distribution along the tube, tube temperature and mercury inlet and outlet temperatures were then taken for each experimental run.

The early experiments on copper-plated nickel tubes were as much directed towards determining whether an upper limit to heat transfer could be reached as to evaluating the heat transfer coefficient itself. The only limit found was when the inside temperature of the tube exceeded the boiling point of the mercury, when a vapour lock occurred and the tube melted locally.

Successful heat transfer work depended on inner surface cleanliness and a good bond between the inner tube and the copper coating. All tubes were internally polished before use, the stainless steel ones being internally fine-ground also to remove roughness from the drawing operation. If care were taken with the cleanliness and



the plating bond of the nickel inner tubes, a tube would give a consistent performance for about 7 to 10 hr., when the outer surface temperature would begin to rise somewhat erratically. This rise might have been due to a failure in the plating bond, to film formation or to corrosion of the nickel tube. Subsequent examination of the inner tube showed considerable evidence of internal pitting, but no conclusive evidence of a plating failure.

Owing to the greater thermal resistivity of the stainless steel, the conduction temperature difference was about  $2\frac{1}{2}$  times that of the nickel tubes, but in spite of the larger corrections involved, the plot of Stanton number against Reynolds number agreed to within 10% with the values for clean nickel.

Short life-tests were made on both nickel and stainless steel inner tubes but, owing to limitations of the apparatus, no runs longer than about 200 hr. were attempted. Within this time scale, stainless steel tubes had a much better life than those of nickel. Runs as short as 35 hr. on nickel tubes gave marked indications of deterioration, as shown by pitting, loss of polish and possible fouling, together with an approximate halving of the heat transfer coefficient.

Similar drops in the heat transfer coefficient were noted with stainless steel tubes after 100 to 200 hours' running, but there was no noticeable pitting or fouling.

#### Possibilities of the heat pump in Canada

The use of the heat pump in different countries and in various circumstances has been described in many papers and articles, but most of the installations described are located in regions where the existing temperature ranges are exceptionally favourable for economic application. Accordingly, the more important stationary installations occur in Switzerland, Central and South America and certain parts of the United States. It is significant that, of approximately 1,400 heat pumps in the U.S.A., the great majority are stated to be situated in a rather narrow band across the continent and are confined to those regions in which the seasonal heating and cooling loads are approximately equal.

The question naturally arises regarding the extent to which the width of this band can be increased in terms of latitude and climate. The following analysis, presented by Prof. E. A. Allcut and F. C. Hooper, University of Toronto, is intended to apply to machines that are employed primarily or solely for the heating of buildings or for other similar purposes where climatic conditions are predominant factors. The applicability of the heat pump under conditions similar to those existing in Canada has been studied by the authors in relation to three typical problems, namely space heating in an industrialised area, space heating in Arctic regions and industrial application in mining operations.

The following factors enter in varying degrees into all of these applications: (1) There must be available a convenient, adequate and reliable source of heat. (2) The temperature difference between the source of heat and its ultimate destination should be as small as possible. (3) There must exist a suitable heating or cooling load (or some combination thereof) and the conditions of operation must be reasonably safe for the personnel and property concerned. (4) There must be available an adequate and suitable supply of power (or its equivalent) at a cost that is appropriate to the circumstances. (5) The equipment must be of such a size, form, weight and simplicity that the resulting capital cost is reasonable. (6) The general economic and industrial environment must be appropriate for installation and maintenance purposes.

Most of the heat pump installations in North America are in urban areas and, as the Province of Ontario is an acute fuel area, an investigation into this problem was considered to be particularly timely. For domestic heating and other similar applications the machine must be small, compact, self-contained and available at low cost. Such machines are almost invariably electrically driven by power obtained from a public utility. In Ontario (and in some other provinces) restricted hydroelectric generating capacity imposes obvious limitations to the possible number of such installations. The nature of the load curve and particularly the timing and duration of the peak loads, both at the generating plants and at the pump itself, are crucial factors which may be decisive. The desirability of the use of low-cost 'off-peak' power is evident.

Although for reasons already mentioned the heat pump is usually regarded as being most suitable for temperate regions, special conditions may extend these limits. In the remote Arctic regions of Canada there is a number of small isolated settlements such as radio stations, trading posts and missions. Transport costs to these stations are extremely high, owing to the great distances involved and the difficulties and dangers of Arctic navigation. In some cases all supplies must be taken by air. It was, therefore, felt that the use of a highly efficient heat pump might be justified on a direct economic basis and might offer additional advantages of an indirect nature. The principal in these cases was that of a suitable heat source. Air, at a design temperature of  $-50^{\circ}\text{F.}$ , is uneconomically low in temperature, and is therefore unsuitable as a source of heat. The ground is also low in temperature and is, moreover, exceedingly difficult to excavate for ground coil installations. There are, however, innumerable lakes, both small and large, in that region and usually the stations are located beside them. The lakes seldom freeze to a depth of more than 8 ft. and consequently a supply of water at about  $32^{\circ}\text{F.}$  is assured.

In some instances the use of sea water is possible.

The extraction of heat from water at this temperature involves freezing to recover the latent heat of fusion. While this procedure presents some mechanical problems, it was felt that the difficulties were offset by the high heat yield (144 B.Th.U./lb.) and the constant and relatively high temperature of the heat supply as compared with environmental temperatures.

A heat pump was designed and built to utilise this source of heat and to be as efficient as was practically possible. It was designed to meet the heating capacity requirements of a small station (130,000 B.Th.U./hr.) and, in this particular installation, the heat was circulated through a water-heating system operated at comparatively low temperatures.

The gold-mining areas of northern Ontario were also selected for study because they are remote from the main industrial areas and have to be supplied with coal, mostly from the United States, at a present price of about \$15 per (short) ton. Substantial reductions in fuel consumption would have obvious economic advantages.

From this study the following conclusions were drawn:

(1) The use of one or more heat pumps, extracting heat from the compressor cooling water and heating the wash water and change house, is definitely economical if sufficient electrical energy is available at the price now prevailing. During periods of very low atmospheric temperature it will probably be advisable to use auxiliary steam heating to improve the load factor on the heat pump.

(2) The heat pump may be used economically for heating (and cooling) other buildings, using the compressor cooling water as a heat source.

(3) The use of heat from mine water or underground rock is only slightly more economical than steam and then only in the larger sizes of heat pump.

(4) The extraction of heat from mill tailings is only practicable in exceptional cases.

(5) The absorption of heat from exhaust mine air is an attractive prospect, but needs further study to produce a workable scheme.

It is realised that the foregoing represents average conditions only and that each mine and locality has its own problems which must be dealt with individually. Nevertheless, it is considered that this analysis gives a reasonably correct appraisal of the situation existing in 1949-50.

#### Acetylene Handling

In the article, 'Handling Acetylene under Pressure in Chemical Reactions,' by S. A. Miller (October 1951), in the second paragraph, page 476, 'At 20 atm., explosive decomposition occurs even in the absence of solid surfaces at  $350^{\circ}\text{C.}$ , but not at  $330^{\circ}\text{C.}$ ,' the temperatures should read ' $450^{\circ}\text{C.}$ ' and ' $430^{\circ}\text{C.}$ '



# The Manufacture of Cemented Tungsten Carbide

By B. E. Berry, M.A.(Cantab.), F.I.M.

(Murex Ltd.)

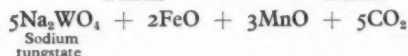
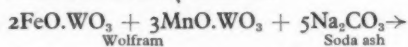
Cemented tungsten carbide, or 'hard metal,' is being increasingly used for a variety of industrial purposes, from dies and tools to textile machinery. Its commercial manufacture is a comparatively new industry and, as is evident from the following article, it involves the co-operation of the chemist, the chemical engineer and the metallurgist.

THE quality of sintered hard metal is materially affected by the chemical and metallurgical processes which are used in the reduction of the tungstic acid from which the tungsten powder is produced. Hence we must first consider briefly the treatment to which the tungsten ore is subjected for the production of tungstic acid.

## Production of tungstic acid

The two principal ores of tungsten are wolfram, which is an iron manganese tungstate, and scheelite, which is a calcium tungstate. The ores are concentrated at the mine and the concentrates, which may contain up to 75%  $\text{WO}_3$ , are then shipped to metallurgical plants in this country, where they are treated.

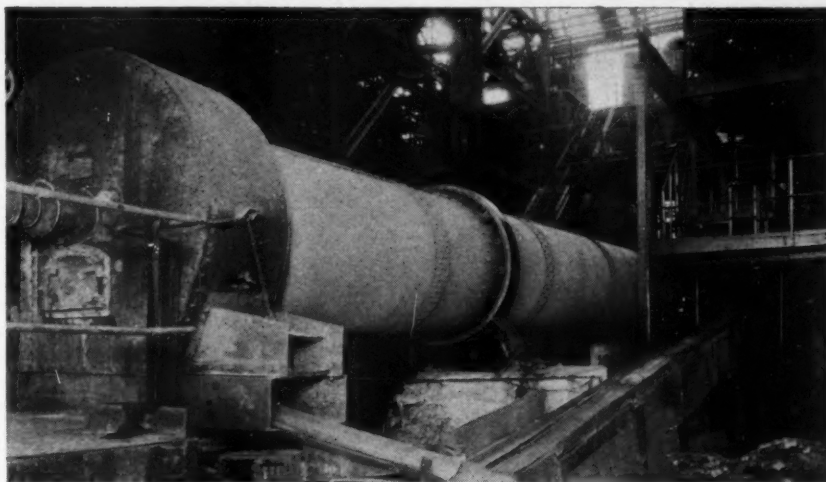
The wolfram concentrates are magnetically separated by means of electromagnets which pick up the magnetic wolfram on to travelling belts, leaving the non-magnetic cassiterite and gänge as tailings. Scheelite, of course, being non-magnetic, is not subjected to this treatment. The separated wolfram is calcined at 800-900° C. to volatilise arsenic and sulphur. The calcined wolfram is then mixed with soda ash and roasted in a furnace or revolving kiln at about 900° C. to convert the tungsten-bearing mineral into sodium tungstate; scheelite may be treated in a similar manner.



The soda roast product is then leached with hot water in a revolving lixiviator, the insoluble residue being filtered off, leaving a solution of crude sodium tungstate.

The crude sodium tungstate liquor, after concentration by evaporation, is cooled down to freezing point to crystallise excess sodium carbonate and impurities such as sodium arsenate and sulphate, which are then removed by centrifuging. The sodium tungstate liquor is treated in a vacuum evaporator to separate the sodium tungstate in crystalline form.

The sodium tungstate crystals are redissolved in water and treated with a solution of magnesium chloride and ammonium chloride to remove the last traces of arsenic. Purified sodium tungstate liquor is then ready for precipitation.



Rotary kiln used for the soda roasting process.

As an alternative to the soda roast process, the separated wolfram may be ground extremely fine and then boiled in an iron vessel with a strong solution of sodium hydroxide. If the wolfram is sufficiently fine, the boiling sodium hydroxide will convert it into sodium tungstate, leaving only small amounts of ore undecomposed. The residue is allowed to settle, and sodium tungstate liquor is decanted off. This liquor may be treated chemically for the removal of molybdenum, arsenic and other impurities.

The pure sodium tungstate liquor prepared by one of these processes is then treated with calcium chloride solution to precipitate calcium tungstate. The conditions under which the calcium tungstate is precipitated affect the grain size of the tungsten metal powder which will ultimately be produced. The strength and the pH value of the sodium tungstate liquor and of the calcium chloride solution, together with the temperature at which precipitation is carried out, must all be carefully controlled to give a calcium tungstate of the required particle size. A weak sodium tungstate solution will give calcium tungstate having comparatively large particle size, while a solution which has a pH value lower than 8.5 will have a similar effect. A high precipitation temperature will give a heavy salt having a large particle size.

The precipitated calcium tungstate is

then filtered from the solution, using a filter press. It is repulped to form a slurry which is run into a heated precipitation vat, where it is treated with hydrochloric acid which reacts with the calcium tungstate and precipitates pure tungstic acid. The conditions under which this treatment is carried out can also affect the grain size of the tungsten powder which will be produced from the tungstic acid. The yellow tungstic acid is washed by decantation and is then filtered off in a filter press. The tungstic acid cake is then placed on shallow trays and dried in a steam-heated oven at 120-130° C. After screening, a representative sample is taken.

This sample is assayed to ensure that the impurities, such as lime, magnesia, silica, etc., are maintained at extremely low limits. The chlorination residue is determined by heating the sample in a stream of chlorine gas and air at a temperature of about 850° C. until all the tungsten has been volatilised. The residue which is left will contain the impurities such as alumina, silica, lime and the alkalis, and should not exceed 0.08-0.10%.

A bulk sample of a few kilogrammes is then converted into tungsten metal powder under the conditions used for the production of very fine grained tungsten powder. This tungsten powder is made into hard metal powder, which is pressed into bars from which test pieces are cut and sintered. These test pieces are polished

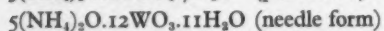
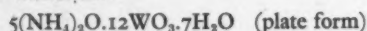
and examined under a microscope to determine the grain size and distribution. They are also checked for hardness and density. Unfortunately it has not so far been possible to formulate a specification for the tungstic acid which will ensure that it is satisfactory for the production of hard metal having the very fine grain size required for the harder grades of tool tips. Hence it is necessary to test each batch of tungstic acid by making hard metal test pieces from it. Work is in progress with a view to establishing a firm relationship between the physical characteristics of the tungstic acid and the grain size of the tungsten metal powder and hard metal produced from it.

#### Ammonium paratungstate

Ammonium paratungstate can also be used for the production of tungsten powder for hard metal manufacture. Generally, the grain size of the hard metal made from this material is coarser than that of hard metal made from fine-grained tungsten metal powder reduced from tungstic acid. The ammonium paratungstate may be produced by dissolving tungstic acid in a solution of anhydrous ammonia in distilled water. The slight insoluble residue is allowed to settle, the supernatant liquor being siphoned off and then filtered for final clarification. The ammoniacal liquor is then evaporated to crystallise the ammonium paratungstate, which is filtered off, preferably on a vacuum filter. The crystals are then dried in a hot air oven below 80° C.

Alternatively, the ammonium paratungstate liquor may be heated to 80° C. and treated with nitric acid (strength 36° Baume) until neutral. The precipitate of ammonium paratungstate is allowed to stand for two days and is then filtered off and dried in a hot air oven.

The ammonium paratungstate crystallises in two forms, the probable formulae of which are:



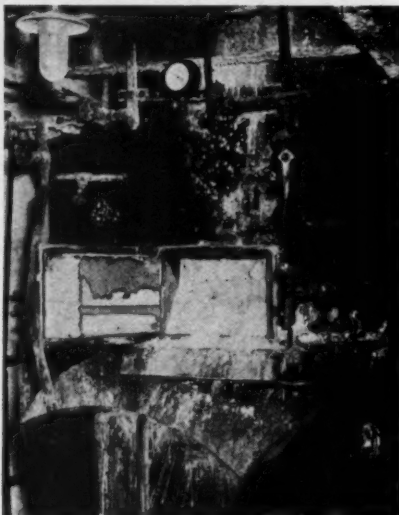
The needle form is generally produced only if the temperature of precipitation or crystallisation is below 50° C.

#### Tungsten metal powder

The grain size of the tungsten metal powder from which the hard metal is produced has a marked influence on the properties of the hard metal. Powder of extremely fine grain size (about 60% less than 0.5 $\mu$  and 90% less than 1 $\mu$ ) is required for the production of hard metal of the highest hardness which is used for machining cast iron, abrasives and other hard materials. For the tougher grades of hard metal which are to be used for mining tools, large tube drawing dies, etc., tungsten metal powder having a coarser grain size, of which about 40% must be under 1 $\mu$ , is used.

It has already been stated that the con-

ditions under which the calcium tungstate and tungstic acid are precipitated influence very largely the grain size of the tungsten metal powder. There seems to be some indication that this influence on the grain size may be due rather to a variation in the surface condition of the particles of the tungstic acid than to the actual grain size of the tungstic acid itself. Consequently certain batches of tungstic acid may be suitable for the production of fine-grained tungsten metal powder, while other batches will tend to form a coarser-grained powder even under reduction con-



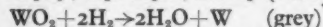
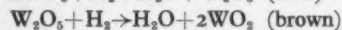
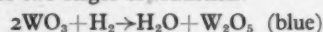
The salt box of the vacuum evaporator, containing the crystalline sodium tungstate.

ditions which would normally produce a fine-grained powder. Nevertheless, the conditions under which the reduction of tungstic acid is carried out have a very definite effect on the grain size of the tungsten powder produced.

In order to produce the very fine-grained tungsten powder for the harder grades of cemented tungsten carbide, the tungstic acid is reduced under hydrogen in small electrically heated tubular furnaces. The tungstic acid is placed in thin layers in heat-resisting steel boats which are fed slowly through these tubes. Pure dry hydrogen is fed into the tubes at the discharge end and is burned at the feed end. Water vapour, which is formed during the reduction, is swept very rapidly out of the tube by the flow of hydrogen.

The tube is maintained at a comparatively low temperature, the tungstic acid being partially reduced to a mixture of the blue and brown oxides of tungsten. These oxides are then given a further reduction under hydrogen in similar tubular furnaces at a higher temperature. This second reduction produces a pure, fine-grained tungsten metal powder having a tap weight of about 28-32 c.c./100 gm., and a grain size in which about 60% is less than 0.5 $\mu$ .

The following reactions take place during these two stages of reduction:



The reduction of tungstic acid to produce the rather more coarse-grained tungsten metal powder, which is required for the tougher grades of hard metal, can be carried out in larger furnaces, since the conditions for reduction are not quite so critical. The tungstic acid is placed in heat-resisting steel trays which are passed slowly through an electrically heated furnace through which hydrogen is circulated. The furnace is maintained at a temperature of between 800° and 1,000° C., and reduction is completed in one operation. The tungsten powder produced under these conditions has a tap weight of 16-17 c.c./100 gm. and a grain size in which 40% is less than 1 $\mu$  and 80% less than 2 $\mu$ .

In Table I typical examples are given showing the tap weight and grain size of various tungsten metal powders and tungsten carbide.

The reduction of ammonia paratungstate to form tungsten metal powder is also carried out in small tubular furnaces. Temperatures up to 1,000° C. are used, the reduction being completed in one stage. The tungsten metal powder produced is rather coarser than that used for the hardest grades of the hard metal but is finer than the tungsten powder used for the tough grades. Tungsten powder produced from ammonium paratungstate is generally considered suitable for those grades of hard metal which contain titanium carbide.

#### Tungsten carbide

The tungsten metal powder, after it has been assayed to determine the loss in hydrogen, which should not exceed 0.2%, is mixed with the correct proportion of

TABLE I  
EXAMPLES OF TAP WEIGHT AND GRAIN SIZE OF TUNGSTEN METAL POWDER AND TUNGSTEN CARBIDE

| Reduction | Tungsten metal powder              |   |                  |                    |                  |                  | Tungsten carbide                   |   |                  |                    |                  |
|-----------|------------------------------------|---|------------------|--------------------|------------------|------------------|------------------------------------|---|------------------|--------------------|------------------|
|           | Tap weight<br>(c.c./<br>100<br>g.) | Grain size<br>(cumulative % by numbers) |                  |                    |                  |                  | Tap weight<br>(c.c./<br>100<br>g.) | Grain size<br>(cumulative % by numbers) |                  |                    |                  |
|           |                                    | Under<br>0.5 $\mu$                      | Under<br>1 $\mu$ | Under<br>1.5 $\mu$ | Under<br>2 $\mu$ | Under<br>4 $\mu$ |                                    | Under<br>0.5 $\mu$                      | Under<br>1 $\mu$ | Under<br>1.5 $\mu$ | Under<br>3 $\mu$ |
| Double    | 31                                 | 58.2                                    | 88.5             | 97.2               | 100              | —                | 17                                 | 68.8                                    | 92.9             | 100                | —                |
| Double    | 29                                 | 48.4                                    | 82.5             | 94.7               | 100              | —                | 16                                 | 63.2                                    | 87.1             | 100                | —                |
| Single    | 17                                 | 29.3                                    | 41.5             | 52.8               | 78.4             | 100              | 14                                 | 50.4                                    | 75.8             | 92.2               | 100              |



Bank of tubular furnaces for reduction of tungstic acid.

fine carbon powder and is revolved in small ball mills for 24 hr. The mixture is then pressed into bars which are placed in carbon boats and fed slowly through electrically heated furnaces in a hydrogen atmosphere at a temperature of 1,500-1,600° C. Alternatively, the mixed tungsten and carbon powder may be placed loose in carbon boats which are then fed through the furnaces. The furnaces usually consist of alumina tubes wound with molybdenum wire, but carbon resistance tube furnaces may also be used. The furnace temperature is automatically controlled by means of a tungsten-molybdenum thermocouple operating a standard controller; temperatures are also checked periodically by means of an optical pyrometer.

The bars of tungsten carbide are removed carefully from the boats, and any loose carbon adhering to the surface is brushed off. The bars are then crushed and milled in small ball mills using hard metal balls until the tungsten carbide all passes a 200-mesh screen. The screen tungsten carbide is then sampled and assayed for total carbon and free carbon. The total carbon should be between 6.05 and 6.2%, while the free carbon must not

exceed 0.1%. It is, however, preferable for the free carbon to be on the high side rather than for the total carbon to be low. The tap weight of the tungsten carbide made from the fine-grained tungsten metal powder is 17-18 c.c./100 gm., while that made from the coarse tungsten powder has a tap weight of 13-15 c.c./100 gm.

#### Tungsten/titanium carbide

Grades of hard metal which are used for machining steel usually contain a proportion of titanium carbide, since this tends to prevent the cratering effect which is obtained when hard metal consisting of tungsten carbide and cobalt only is used for this purpose. The titanium carbide is frequently added to the mixture of tungsten carbide and cobalt in the form of a double carbide of tungsten and titanium.

This tungsten/titanium carbide is prepared by heating a mixture of titanium dioxide, tungsten metal powder and carbon to a temperature of 2,200° C. The powders are mixed together by prolonged ball-milling and are packed firmly into a carbon crucible. This crucible is placed in a high-frequency furnace coil and heated gradually to the required temperature. The carbon reduces the  $TiO_2$

and forms titanium carbide, at the same time carburising the tungsten metal powder. Alternatively, the mixture of titanium dioxide, tungsten metal powder and carbon may be placed in a carbon boat and carburised at 2,200° C. in a carbon tube furnace. The two carbides form a mixed crystal which is generally considered to be more satisfactory for incorporation in hard metal than pure titanium carbide. It has been shown frequently that hard metal made with pure titanium carbide tends to have a slightly soft centre after sintering.

The mixed carbide is then crushed, ball-milled and screened on a 200-mesh screen and is sampled and assayed for total carbon and free carbon. For a carbide containing equal proportions of titanium carbide and tungsten carbide the total carbon should be about 13% with not more than 1% of free carbon.

#### Hard metal powders

The composition of the hard metal powders will vary very widely according to the purpose for which the hard metal is required. For machining cast iron and materials of an abrasive nature fine-grained tungsten carbide will be used, while the proportion of cobalt will vary between 4 and 10%, depending on the toughness which is required. For large drawing dies, mining tools and other applications where considerable toughness is required, coarser grained tungsten carbide will be used and the cobalt content will vary from 6 to 30%. For grades of hard metal for use in machining steel, titanium carbide may be added up to about 15%; tantalum carbide is also used.

The composition and physical characteristics of some typical hard metal compositions are shown in Table 2.

The various carbides and the cobalt metal are weighed out in their correct proportions and are placed in small ball mills using hard metal balls. Acetone is added and the mills are either revolved or vibrated for several days to ensure the perfect mixing of the cobalt with the hard carbides. While the grain size of the tungsten carbide in a sintered hard metal is primarily determined by the grain size of the tungsten metal powder which is used, this grain size can also be affected to some extent by the period of wet

TABLE 2. COMPOSITION AND PHYSICAL CHARACTERISTICS OF TYPICAL HARD METAL COMPOSITIONS

| Composition  | Hardness<br>(V.P.N.) | Density<br>(g./c.c.) | Transverse<br>rupture<br>(lb./in. <sup>2</sup> ) | Compressive<br>strength<br>(lb./in. <sup>2</sup> ) | Coefficient<br>thermal<br>expansion<br>(per °C.) | Thermal<br>conductivity<br>(cal./cm. <sup>2</sup> /<br>cm./sec./<br>°C.) | Sp. ht. | Electrical<br>resistance<br>at 20°C.<br>(microhms<br>/cm. <sup>2</sup> ) |
|--|----------------------|----------------------|--|--|--|--|---------|--|
| 1. Cobalt 4%. Fine-grain tungsten carbide 94% ..                                 | 1800                 | 15.05                | 160,000  | 780,000  | $5 \times 10^{-6}$                               | 0.20   | 0.05    | 21.0   |
| 2. Cobalt 6%. Fine-grain tungsten carbide 94% ..                                 | 1650                 | 14.80                | 220,000  | 750,000  | $5 \times 10^{-6}$                               | 0.19   | 0.05    | 20.0   |
| 3. Cobalt 6%. Coarse-grain tungsten carbide 94% ..                               | 1400                 | 14.80                | 260,000  | 680,000  | $5 \times 10^{-6}$                               | 0.19   | 0.05    | 20.0   |
| 4. Cobalt 10%. Coarse-grain tungsten carbide 90% ..                              | 1300                 | 14.35                | 290,000  | 620,000  | $6.0 \times 10^{-6}$                             | 0.16   | 0.05    | 18.0   |
| 5. Cobalt 7%. Titanium carbide 5%. Medium-grain<br>tungsten carbide 88% .. .. .  | 1500                 | 13.35                | 210,000  | 630,000  | $5.5 \times 10^{-6}$                             | 0.15   | 0.05    | 25.0   |
| 6. Cobalt 8%. Titanium carbide 15%. Medium-grain<br>tungsten carbide 77% .. .. . | 1500                 | 11.10                | 195,000  | 590,000  | $6 \times 10^{-6}$                               | 0.09   | 0.06    | 43.0   |



milling and the ratio of the ball charge to the weight of powder in the mill.

The cobalt is generally added as fine cobalt metal powder, although some manufacturers consider that more even distribution is obtained by adding the cobalt in the form of cobalt oxide. During the wet milling process the cobalt oxide will tend to be very evenly dispersed throughout the tungsten carbide. The cobalt oxide will subsequently be reduced to cobalt metal during the furnace treatment to which the powder is subjected after milling.

After milling, the powder is allowed to settle and the acetone is siphoned off, the last traces being removed under vacuum. The damp powder is then placed in small heat-resisting steel boats and is fed slowly through small tubular furnaces under hydrogen at a temperature of about 600° C. This furnace treatment reduces any slight traces of cobalt oxide which may have formed during the milling; if the cobalt has been added as oxide, this treatment will reduce the oxide to metal.

Owing to its extreme fineness, the hard metal powder is difficult to press and tends to produce cracked and laminated bars. In order to improve its pressing properties the powder is mixed with a solution of paraffin wax in carbon tetrachloride and is warmed slightly to evaporate the carbon tetrachloride, thus leaving the particles of powder coated with a thin film of paraffin wax. Alternatively, some manufacturers prefer to treat the powder with a solution of camphor dissolved in ether or in petrol, while others use ethylene glycol. After screening, the waxed powder is sampled and a test bar is pressed; test pieces are cut and sintered.

One of the test pieces is fractured and the surface of the fracture is examined; sound hard metal of requisite strength has a slightly rugged fracture, while a fracture of very smooth and silky appearance suggests a tendency to brittleness at times unavoidable in the very low cobalt grades. The other test piece is examined for density and hardness and is then polished for micro-examination. One face of the test piece is ground flat on a 400-mesh metal bonded diamond wheel, about 0.008 in. being removed to expose the inner structure. A further 0.002 in. is then removed by grinding with a 750-mesh steel bonded diamond wheel at right angles to the direction in which the previous grinding was carried out. The specimen is then polished, again at right angles, on a revolving leather disc impregnated with superfine diamond powder in oil.

The polished test piece is first examined at 100 magnifications to determine porosity. At 1,500 magnifications micro-porosity and intergranular porosity can be seen, and the distribution of the cobalt round the grain boundaries is also in evidence. The test piece is then etched in alkaline ferricyanide, which etches the cobalt bond, leaving the tungsten carbide unattacked. The grades containing ti-

tanium carbide are etched in nitric and hydrofluoric acids, which attack the cobalt bond and render the titanium phase slightly pinkish. The grain size of the tungsten carbide can be checked against standard charts. If porosity and grain size are satisfactory, the powder is passed for used.

### Pressing and shaping

The waxed hard metal powder is then pressed in steel or carbide lined dies at a pressure of 10 tons/sq. in. Where large numbers of pieces of a particular shape are required, automatic or semi-automatic presses may be used. Wire-drawing die pellets may be pressed ready cored in a steel die in which the back relief is formed on the baseplate of the die, while the entrance angle and bore are formed on the punch. A weighed quantity of powder is placed in the die and the assembly is pressed by means of a hand-operated hydraulic press. The powder is also pressed into bars in collapsible dies on a large hydraulic press. Cylindrical briquettes can be pressed under hydrostatic pressure, and recent developments have provided a means of extruding rods and tubes of hard metal, thus widening enormously the field of application.

The briquettes and pellets are then packed in carbon powder in heat-resisting steel boats and are passed slowly through long presintering furnaces in a hydrogen atmosphere. The paraffin wax is vaporised in the first sections of the furnace and the briquettes are finally heated to about 750° C. to make them firm enough to handle and machine. The bars and briquettes are then machined and ground to the required shapes, due allowance being made for the shrinkage which will have taken place on sintering.

The tips and pellets are all carefully checked for dimensions prior to sintering, as any which are incorrect at this stage can be crushed up and re-used. Pieces of presintered bar, etc., which are left after cutting the tips are also crushed and screened and incorporated in new batches of powder. As each individual machine is provided with a separate dust-collecting unit, it is also possible to collect and re-use the dust from the machining process if care is exercised to ensure that each grade is kept separate.

### Sintering

The sintering operation is carried out in electrically heated furnaces which may be either of the alumina tube type with molybdenum wire windings or of the carbon tube resistance type. These furnaces are arranged for accurate temperature control, as the sintering temperature of the various grades of hard metal powder is very critical.

The shaped hard metal pieces are packed in carbon powder in carbon boats. These boats are then passed slowly through the sintering furnace at a steady rate by means

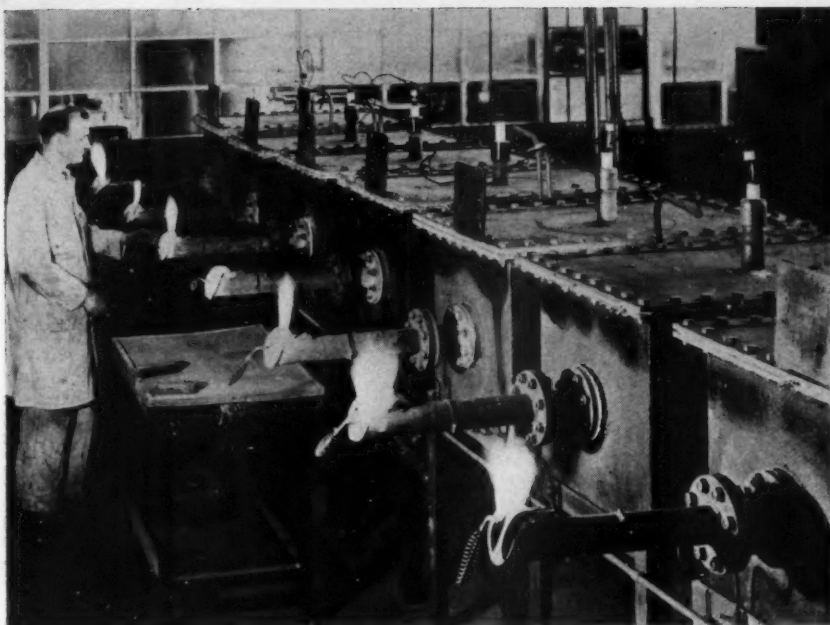
of a mechanical pusher. After passing through the furnace, the boats enter a water-cooled zone and are finally taken out of the furnace when cold. The whole operation is, of course, carried out in an atmosphere of hydrogen. The sintering temperature varies from about 1,300° C. for grades containing 20% cobalt to nearly 1,600° C. for titanium carbide grades. During the sintering process the cobalt metal becomes molten and dissolves about 14% of tungsten carbide, all but 1% of which is reprecipitated on cooling. The hard metal pieces shrink about 20% on sintering and attain their final hardness and density.

Instead of packing the tips in carbon powder for sintering it is sometimes considered advisable to use alumina powder. If, however, alumina powder is used without the admixture of small amounts of carbon powder there is a very strong tendency for the surface of the tip to be decarburised. Tips sintered in alumina powder generally show a slightly higher density. On the other hand, the carbon packing tends to minimise distortion and bending during sintering, probably owing to a more even heat distribution. Hard metal tips which have been packed in carbon powder for sintering tend to have a slightly harder surface layer, possibly due to a slight migration of the cobalt away from the surface, or the formation of small amounts of cobalt carbide in these surface layers. This makes the surface more resistant to abrasion, an effect which is particularly noticeable in percussion drill stems if the periphery of the carbide insert has not been ground prior to drilling. For the first one or two holes drilled the gauge loss per foot drilled is negligible, but after the first skin has been worn the gauge loss shows a normal figure of 0.0005-0.0015 in. per foot drilled.

Free tungsten is sometimes added to hard metal powder to obtain an increased resistance to abrasion. This free tungsten may, to some small extent, become incorporated in the cobalt bond, but appreciable quantities react with the tungsten carbide to form the lower carbide  $W_2C$  which, while more brittle, is harder and more wear resistant than the saturated carbide. If such grades are sintered in carbon powder, the surface layers will be free from  $W_2C$ , owing to reaction between this compound and the carbon packing.

### Hot pressing

The hot pressing method is used for making large pieces of hard metal and also for pellets of intricate bore shape. A graphite mould is machined to the dimensions of the required sintered hard metal piece, while a graphite core pin, formed to the shape of the bore, is placed centrally in the mould. The graphite plunger slides on the core pin in the mould. A weighed quantity of hard metal is poured into the mould and the plunger is placed in position. The mould is then



**Bank of carburising furnaces.**

(This and the other photographs are published by permission of Murex Ltd.)

set up in a high-frequency coil and a hydraulic press head is arranged to apply pressure to the plunger, while the mould and the hard metal powder are being heated to sintering temperature by means of the high-frequency current. As the sintering temperature is approached, the powder begins to shrink and the pressure applied causes the plunger to move downwards, thus compressing the powder as it sinters. When sintering is complete the current is switched off and the hard metal piece is allowed to cool down in the mould. The carbon mould is then broken away and the hard metal pellet is cleaned and finally inspected.

In the hot pressing method all the shrinkage takes place in the direction of pressing, so that it is possible to maintain a very close tolerance on the bore and diameter. As an alternative to the use of the high-frequency current the graphite mould itself may be used as a resistor by passing a high current through it. The quality of the hard metal made by the hot pressing process is similar to that produced by cold pressing and sintering.

All hard metal pieces, whether made by the cold pressing and sintering process or by the hot pressing process, are inspected very carefully for cracks, chips and flaws. The dimensions are measured accurately, and hardness and density tests are carried out on a percentage of the pieces produced. The hardness figure serves as a check on the sintering conditions.

#### **Dies and tools**

In the die-making department tungsten carbide pellets are ground on the periphery and fitted into steel cases, either by brazing with silver solder, bronze or copper, or by

shrinking into the heated case. An interference fit of a few thousandths of an inch is left so that the steel case, which on heating expands more than the carbide, will hold the pellet firmly on cooling. The bore of the die is then ground to shape using diamond wheels, and is finally lapped with diamond powder in oil to give a highly polished surface having a surface finish of about two micro-inches. As an alternative to brazing, a newly developed process makes it possible to treat the surface of the sintered hard metal to render it amenable to the use of ordinary solder.

Metal-bonded diamond wheels, as well as vitreous and Bakelite-bonded ones, are being used more and more for the grinding and finishing of hard metal dies and other parts. As skill in the use of these wheels improves, the initial high cost is more than offset by the life obtained and the finish which it is possible to impart to the carbide surface.

Hard metal tips for lathe tools are surface ground and are then copper brazed or silver soldered into a recess which has been milled in the carbon steel tool shank. The shank and tip are then finally ground to the required dimensions. The cutting edge of the tip may be diamond lapped, although this is not always considered necessary.

Hard metal inserts may also be used for tipping rock drill stems. The drill stem is forged and a slot machined in the head. A hard metal insert is then brazed in this slot, care being taken to avoid any undue stress in the carbide. The head is then ground to final size, and the stem is ready for use. These carbide-tipped stems are finding increased use in the mining areas of

the world, and their life is being continually increased as the quality of the carbide and the methods of assembly are improved.

#### **Conclusion**

The production of hard metal has developed very considerably during the last ten years, both in the size of piece which can be produced and the intricate shapes which it is possible to form.

The development of the extrusion process, referred to above, has extended very considerably the range of products which can be made. Hard metal tubes having a bore of 0.020 in. and a wall thickness of 0.010 in., which are used as guides in the textile industry, can now be produced by this process. Improvements in pressing and sintering technique allow the production of the large flat tips of special shape which are required in the pottery trade, while it is also possible to produce the long cylindrical pieces which are required for rolls for the rolling mills.

Industry continues to make more and more demands on the hard metal manufacturer and to ask for even closer tolerances. In meeting these demands, new techniques are developed, new problems are solved and further fields of application are opened up. This ever-widening sphere unites hard metal manufacturers and users alike in that close collaboration which is necessary for the rapid development of this new and expanding industry.

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#### **Recent publications**

**Chemical engineering services.** The design, engineering and building of oil refineries and plants for the production of chemicals from oil and other raw materials is carried out by Petrocarbon Ltd., whose services are outlined in a well-illustrated brochure. It is stated that the company has all necessary facilities and equipment for the detailed examination of economic and technical possibilities and prospects, research and development work, and the design, engineering, construction and running-in of plants.

**Acid-resisting construction.** The construction of acid-resisting linings for reaction vessels, acid storage tanks, scrubbers, chemical plant and flooring is described in a leaflet from Acalor Ltd. Chemical-resistant brick and tile linings are supplied by the company, who also manufacture a large variety of jointing cements for bonding the linings. The firm also supply neutralising and settling tanks as well as complete schemes for treatment of effluents.

**Nickel.** A recent issue of 'The Nickel Bulletin,' published by The Mond Nickel Co., Ltd., gives tables and charts of the physical and mechanical properties of pure nickel.



# Lord McGowan Surveys a Chemical Century

IF Dalton were still alive, he would not be disappointed with the record of British chemists in the past hundred years, though he was never given to praising lightly. That was the opinion of Lord McGowan, K.B.E., honorary president of Imperial Chemical Industries, when he delivered the seventh Dalton Lecture in Manchester on September 27, 1951.

Lord McGowan decided to confine himself, so far as was just and reasonable, to the contributions made by British chemists during the period since 1851. After touching on their achievements in the field of pure chemistry, he went on to remark on the rapid advance in industrial chemistry. Today, chemistry touches almost every aspect of our lives, individual and national. In peacetime, chemical research was the whetstone that put the fine edge on our competitive efficiency as an industrial nation, and in war, was one of the most important factors in military security.

This was not the case in 1851, when civilisation was built round wood and iron, with no man-made materials for any specific purposes. Some, like polystyrene and polyvinyl chloride, were known or soon to be known, but they were simply laboratory curiosities. The atom was regarded as the smallest particle of matter and its physical reality was in some doubt. However, the foundations of modern knowledge were being laid. The increase of that knowledge may be gauged from the fact that the whole of chemistry as known in 1851 could be published in 8,000 pages taking 2½ ft. of shelf space, whereas today 61 volumes, taking 11 ft. of shelf space, were necessary for organic chemistry alone. Sixty elements were known in 1851 compared with 92 today. There were probably half a million chemical compounds today as against 20,000 in 1883. The chance that any new chemical compound made today would be technically useful was, at most, about 1 in 50.

## Medicine

Lord McGowan went on to review the application of chemical knowledge to medicine and surgery, leading to chemotherapy, which had introduced a new and great era of medicine. Organic chemistry, having made possible the isolation of the active principles of many vegetable drugs, had gone on to open up a field of new synthetic drugs.

Listing a few examples of British successes in this field, Lord McGowan referred to Fleming's discovery of penicillin, to British contributions to the development of the sulphonamides, anaesthetics and analgesics. Chemistry supplied the anaesthetist with his materials, and the anaesthetist, in his turn, had made possible the real advance of surgery.

From surgery the speaker turned to vitamins, of which nothing was known a hundred years ago, though vitamin defi-

ciency diseases were only too well known. Other remarkable advances had been made in synthesising hormones, anti-malarials and drugs to control leprosy.

## Dyes

Lord McGowan recalled Perkins' discovery of the first coal-tar dye. However, this was preceded by Faraday's discovery of benzene—the starting point of the synthetic dye industry and of such substances as aspirin, and saccharin. In the early days of the British dyestuffs industry discovery succeeded discovery: azo dyes, the first acid dyes for wool, and aniline black. Afterwards, the lead was lost to the Germans. Today Britain had recovered the lead in dyestuffs and was producing more than 2,000 different dyes from indigenous materials.

## Plastics

Plastics was another child of the energetic organic chemical school fostered by the dyestuffs industry. Plastics were unknown commercially in 1851. Thomas Hancock discovered how to make ebonite and developments with rubber were followed by the invention of linoleum. Notable developments with cellulose were contemporary and a horny material made of nitrocellulose mixed with camphor was exhibited in 1862. Later developments produced celluloid, leather cloth and, finally, the modern plastics industry was born in the U.S.A. Admitting the American genius in plastics, nevertheless some of the greatest advances of the last two decades had been made in the U.K. *Perspex* was a British discovery. So, too, was polythene, the result of a splendid and courageous piece of research on high-pressure reactions.

## Synthetic fibres

From plastics it was a short step to man-made fibres, first developed from molten glass. Swan, of electric light fame, invented a process of squirting nitrocellulose solution through a die into a coagulating bath of water and alcohol, and this has become the basis of synthetic fibre production. In 1893, Cross, Beadle and Bevan made viscose from cellulose, which invention was followed by acetate rayon, first made at Spondon, near Derby. Great developments in the perfection of protein fibres had been made of recent years, and truly synthetic fibres were being made today. Their manufacture resulted from chemical investigation of long-chain molecules. *Terylene*, the polyester fibre, now due to be commercially produced, and *Ardil*, the groundnut protein fibre, were other great British advances in synthetic fibres.

## Agriculture

Lord McGowan then turned to contributions of chemists to agriculture, begin-

ning with the treatment of bones with dilute sulphuric or hydrochloric acid to make superphosphates, followed by the arrival of nitrate of soda from Chile and then the fixation of the inexhaustible nitrogen of the atmosphere.

Chemistry had elucidated problems of soil structure and uncovered secrets of plant growth. Another great contribution had been in the field of crop protection with weedkillers, plant growth regulators and insecticides. Mention was made of those selective weedkillers, which would destroy weeds, leaving crops unharmed, of a new fungicide, developed in England, which showed promise in controlling mouldy rot of rubber trees, of DDT and of the new *Gammexane*, Gamma BHC, a product of British research and, in some cases, more effective than DDT. British soil alone could yield £180,000,000 worth more of food per annum if plant protection remedies were fully applied. Before concluding this part of his talk, Lord McGowan referred briefly to the new systemic insecticides, which could be absorbed harmlessly by plants, while rendering their sap poisonous to sucking insects.

In conclusion, Lord McGowan made brief references to soap and synthetic detergents, to chemical refrigeration, to water softening and to synthetic perfumes. He ended by mentioning that since the institution of Nobel Prizes Britons had won the chemical prize seven times.

## Correspondence

To the Editor,  
INTERNATIONAL CHEMICAL ENGINEERING.

### South Africa's petrol-from-coal plant

Dear Sir,—It is with interest that we have read your article in the May issue. We should like to draw your attention to the fact that the contents of the article do not quite agree with the actual facts in so far as the order to build the South African petrol-from-coal plant was not given to the M. W. Kellogg Co. alone, but was distributed to several firms. The gas production and gas purification plant will be built by Lurgi, Gesellschaft für Wärmetechnik, Frankfurt, and it is intended to purify the gas according to the *Rectisol* process developed by Ruhrchemie-Lurgi.

One-third of the synthesis plant proper will be built according to the Ruhrchemie-Lurgi process, which is based on long experience of the German team which has been working on the development of the Fischer-Tropsch process since the beginning of the 'thirties.

DR.-ING. H. NEDELMANN,  
Fachverband Kohlechemie  
Essen, Germany.



# Chemical and Engineering Research in Australia

A considerable number of chemical engineering and chemical investigations are organised and directed in Australia by the Commonwealth Scientific and Industrial Research Organisation which was set up in 1949 to take the place of the Council for Scientific and Industrial Research. The Organisation has recently published its second annual report which records work undertaken up to the middle of 1950. From it is taken the following summary of the progress of investigations in chemical engineering, organic chemistry, ore-dressing, fuel technology, physical metallurgy, ceramics and minerals utilisation. Work described in the Organisation's first annual report was noted in **INTERNATIONAL CHEMICAL ENGINEERING**, September 1950.

## Chemical engineering

**W**ORK has continued on a number of projects designed to provide fundamental information of importance for developing methods of chemical engineering design and new methods of processing. Progress has been made in the installation and instrumentation of a wide variety of pilot-scale processing equipment. The units already installed have been used extensively for pilot-plant studies by officers of the Division and by the technical staffs of a number of industrial organisations.

The Section's work, particularly applied research on industrial adsorption processes and fuel utilisation, has expanded considerably. The Section has also continued its advisory service to chemical industry, and has provided facilities for the design and construction of equipment for other sections of the Division.

## Physical properties of fluids at high pressures

The laboratory for this work is located in the Chemical Engineering Department, University of Sydney, and was first occupied during 1949. When the necessary high-pressure equipment is installed, fundamental studies will be made of the physical and thermo-dynamic properties of compressed fluids. During the course of this work the Section will collaborate with similar English and American laboratories in preparing tables and charts of physical and thermo-dynamic properties of industrially important fluids and fluid mixtures.

Pending the installation of the high-pressure equipment, experimental work has been undertaken on two topics which, while relevant to the investigation, do not require extensive high-pressure equipment. These are:

(1) Determination of second virial coefficients for gases and vapours by a procedure designed to permit greater accuracy of measurement than that obtainable by existing methods. The second virial coefficient of a gas or vapour, which represents the initial departure of the gas from the ideal behaviour, is important at relatively low pressures.

(2) A study of the effect of solvation on the dipole moments of molecules in solution in hydrocarbons under pressure.

## Distillation

The study of equilibrium conditions for the liquid and vapour phases of binary mixtures has been continued and data have been obtained for the systems carbon tetrachloride/cyclohexane at 70°C., benzene/*n*-heptane at 60°C. and 80°C., and ethanol/*n*-butane at 760 mm. Other systems are being studied with the ultimate aim of predicting liquid-vapour equilibria from a knowledge of the structure of the pure components of binary and ternary mixtures.

## Solvent extraction

An experimental spray column has been constructed and used to measure the rate of extraction of benzoic acid from aqueous solutions by benzene, for a range of drop sizes and velocities. These results will be combined with previously determined diffusion coefficients for benzoic acid in water and in benzene, in an attempt to determine diffusion film thickness. The object of this work is to develop methods for designing industrial equipment for continuous solvent extraction. It is being done by an officer working at the Chemical Engineering Department, University of Sydney.

## Development of adsorption processes

At present there is no satisfactory industrial process analogous to fractional distillation for the separation of complex mixtures such as sea-water, fermentation process wastes, sewage, high boiling point mixtures of hydrocarbons, fats, oils and waxes. However, the components of mixtures of this kind can be selectively removed by adsorption on finely divided carbon or other adsorbents from which they can subsequently be removed (desorbed). This method of fractionation should be commercially attractive if it can be developed into a continuous process in which desorption can be effected without the use of organic solvents.

The Section has made considerable progress in the development of two adsorption processes, namely a vat process having a large throughput, and a column process having a smaller throughput but giving more effective fractionation. Success was also achieved in modifying the surface of adsorbents to render them more selec-

tive, and in using froth flotation to obtain counter-current flow. These advances have made it possible to operate these processes effectively on a continuous basis.

The vat process has been operated continuously for some time, and it has been demonstrated that large quantities of liquid can be processed with negligible losses of adsorbent and flotation agent. In developing the continuous column process, simple equipment has been evolved which has the advantages of effective fractionation and ease of operation.

Work has continued on the development of improved adsorbents, particularly modified carbon adsorbents, and a study has been made of the co-adsorption on activated carbon of insoluble non-electrolytes, particularly flotation reagents, and of soluble weak organic electrolytes.

The Section is also developing an industrial process for using ion exchange resins, which differs from the conventional procedure in that adsorption occurs as a result of the selective short-range adsorptive forces, and the electrostatic forces are utilised only during the desorption stage.

## Study of fine particles

To study the behaviour of fine particles suspended in gas or liquid media, methods were developed for preparing uniform spherical particles and measuring their size. During the year a comparison of the flow of fluids through suspensions and beds of these particles was commenced; this was made possible by the development of extremely thin filter membranes with negligible resistance to fluid flow. The aim of the work is to obtain basic information which can ultimately be applied to such processes as filtration, sedimentation, thickening and dust and fume removal.

## Unit operation

Pilot-scale chemical plant equipment installed in this laboratory has been used during the year by officers of the Division for the extraction of alkaloids from Australian plants, for the extraction of wool wax alcohols, and for the purification by fractional distillation of various organic intermediates. This equipment has also been made available to private companies for work on the spray drying of various materials, for vacuum and pressure filtra-

tion, and for the digestion and pulping of cellulosic materials.

### Wool wax

Current investigations aimed at elucidating the composition of this material, which is essentially an ester wax, include: (1) development of physical means for resolving the ester mixture and determining the composition of the fractions; (2) construction of a modified spinning-band distillation column for low-pressure fractionation of the aliphatic alcohols; (3) further investigation of the molecular structure of lanosterol; and (4) further study of the alkaline hydrolysis of wool wax.

### Sugar cane wax

This is potentially available in quantity by solvent extraction of the filter muds from the sugar mills. The crude wax is soft and dark, and, before it can find extended use, needs to be hardened by refining and bleaching. Refining is usually accomplished by solvent treatment. It has now been shown that the softer and acidic components of the wax may be removed by heating under reduced pressure, and that the residual hard dark neutral wax closely resembles the product obtained by solvent treatment. This hard wax can be bleached oxidatively with chromic acid to give hard pale yellow acidic waxes. Steps have been taken to patent this refining process.

### Alkaloids

The investigation of stock poison plants in collaboration with the Division of Annual Health, which commenced last year as a minor problem concerned with the isolation of the alkaloid lasiocarpine from *Heliotropium europaeum*, was extended. Difficulties encountered during the isolation of lasiocarpine led to a more detailed examination of *H. europaeum*, and it is now clear that the plant contains a complex and variable mixture of tertiary bases and their N-oxides. A quantitative method for the determination of these alkaloids is now being sought in order to study seasonal and local variations.

It now seems desirable to examine other native plants likely to contain the hepatotoxic pyrrolizidine alkaloids such as occur in *H. europaeum*. Further, an attack is to be made on the group of plants causing locomotory disturbances in animals, and a survey is in progress of the Australian Cycadaceae for toxic pseudo-cyano-genetic glucosides. The majority of Cycadaceae so far tested have given positive results and the macrozamin previously isolated from *Macrozamia reidleyi* and *M. spiralis* has been found in *M. miquelii*.

A number of Australian commercial timbers are reputed to produce adverse physiological effects in man, the effects being most evident when the timber is in a finely divided condition, as in sawdust. Two such timbers are being examined with the object of isolating and identifying the

active principles: *Balanops australiana*, which causes nasal irritation and sneezing, and *Dysoxylon muelleri*, which causes severe irritation of the eyes, nose, throat and lungs. In each, extraction has yielded 1 to 2% of a colourless solid. That from *Dysoxylon* is a complex mixture separable by chromatographic methods into a number of fractions, some of which have been obtained in crystalline form. The material extracted from *Balanops australiana* is crystalline and appears to be triterpenoid. The physiological aspects of the *Dysoxylon* investigation have been undertaken by Prof. Sir Stanton Hicks of the Physiology Department, University of Adelaide.

Alkaloids from members of the family Rutaceae were further studied. Leaves of *Acronychia baueri* yielded eight alkaloids, four of them acridine derivatives and four furanoquinolines. The former were identified as meliocopine, meliocopicine, melicopodine, and 2, 4-dimethoxy-N-methylacridone, this being the first recorded occurrence of acridine derivatives in the plant kingdom. Three of the furanoquinoline alkaloids were identified as acronyridine, skimmianine and kokusaginine. The fourth has been named acronidine.

Much structural work in this field was done in collaboration with members of the Chemistry Department of the University of Melbourne. Medicosmine and acronidine (from *Medicosa cunninghamii*) were shown to be respectively the mono- and dimethoxy derivatives of tetracyclic dimethylpyranofuranoquinolines, and the first recorded members of this class. Again, in collaboration with the Chemistry Department, the structures of three alkaloids from *Pentaceras australis* were established as derivatives of a base cantine (itself still unknown), viz., 11-canthinone, 12-methoxy-11-canthinone, and 13-thio-methyl-11-canthinone. Alkaloids from other Rutaceae species under examination include those from *Lunasia amara* and *Micromelum pubescens*.

Work has commenced on the chemistry of the highly toxic and vesicant alkaloid cryptopleurine from *Cryptocarya pleurosperma* (Lauraceae) and the nature of the functional groups has been established. Examination of other *Cryptocarya* species resulted in the isolation from *C. hypospodia* of an alkaloid not identical with cryptopleurine.

### Plastics

(1) *Kinetics of the phenol-formaldehyde condensation.* The special phenols required for this work were not available commercially and were prepared. Mesityl (2, 4, 6-trimethylphenol) and pentamethylphenol were readily obtained, but the synthesis of prehnitenol (2, 3, 4, 5-tetramethylphenol) was time-consuming and its purification difficult. Other preliminary work completed included the measurement of the solubilities in dioxane-water mixtures of the reagents, products and catalysts of

the reaction to be studied. Suitable analytical methods for following the reaction under alkaline conditions were also tested.

Experiments showed that in the presence of the inert phenol, mesitol and, under mildly alkaline conditions, the transformation of formaldehyde accorded with a normal Cannizzaro reaction. With the reactive phenol prehnitenol present, the Cannizzaro reaction was by no means suppressed, and was still responsible for 30 to 90% of the total loss of formaldehyde. Elucidation of the prehnitenol-formaldehyde reaction is, therefore, not at all simple.

(2) *Adhesives of tannin-formaldehyde.* Batches of this adhesive were prepared from mimosa extract and used by the Division of Forest Products to determine its behaviour in a roller spreading machine. The adhesive gave good dry and wet strengths in the resulting plywood, but as a warm-press adhesive it had a rather short working life.

### Heterogeneous catalysis

Information on the mechanism of catalytic dehydration by a thoria catalyst is being sought through measurement of the rates of adsorption of gases by the solid catalysts. This is an extremely rapid process and special apparatus has been developed for the purpose. At the same time, information is being obtained on the structure of the particularly active thoria catalyst obtained by pyrolysis of thorium oxalate.

### Other investigations

The reaction between glycerol and hydriodic acid, a method for the quantitative estimation of glycerol, was studied. The mechanism of the reaction, which gives a quantitative yield of the product isopropyl iodide only when an aliphatic organic acid is used as a catalyst, was determined.

Analysis of samples taken of the Kerguelen cabbage (*Pringlea antiscorbutica*), a plant peculiar to the Kerguelen-Heard Island group, has shown that it has a high content of vitamin C.

At the request of the Division of Animal Health and Production the compound phenylpantothenone, a possible antagonist in bacterial metabolism, was synthesised.

### Ore-dressing investigations

Ore-dressing investigations are being carried out in co-operation with the Kalgoorlie School of Mines. Much attention was given to the testing of various mine dumps for the possibilities of extracting the gold from them by cyanidation. A number of requests were also received involving tests aimed at the determination of the best method of treating ores; such requests came from Marble Bar, Calyerup Creek, Cordoroy, Gabanintha and several other localities where gold is found. The facilities of the laboratory were also in



considerable demand for infrasing tests of mill products such as slimes as a guide to improving the metallurgical treatment adopted by the large mines of Kalgoorlie and elsewhere.

At Melbourne, where work is carried out in co-operation with the Department of Mining of the University, 20 reports were issued. Of these, six relate to beach sands, five to gold ores and tailing dumps, and the remainder to ores of tin, lead, copper and bismuth, and to iron oxides, brown coal and talc. Twenty-nine investigations, mainly relating to the treatment of gold and tin ores and dumps, and to the recovery of tin and monazite from beach sands, were in hand but incomplete at the end of the period under review.

The electrostatic separator designed and constructed in the laboratory has been modified by installing sheet electrodes, one on each side of the stream of falling particles. With these charged at opposite polarities, greater separating efficiency was attained and satisfactory separation of zircon from rutile was achieved. These are two of the valuable minerals which may be recovered from beach sands which occur along the eastern Australian coast and elsewhere. Combinations of magnetic and electrostatic methods of separation have also been used to recover monazite and cassiterite from beach sand concentrates.

Difficulties associated with the acid leaching and recovery of bismuth from an ore occurring in an isolated and arid region have been investigated. A lead ore from western New South Wales and a copper ore from Queensland each contained appreciable quantities of oxides and carbonates of the valuable metals. Further work is in progress designed to establish conditions for good recovery of such minerals by flotation concentration.

#### Flotation investigations

In flotation the surface of a mineral under water is modified (conditioned) so that a bubble of air may make contact with it and buoy it up into a froth. By measuring the amount of a soap (sodium cetyl sulphate) adsorbed on a known area of cassiterite surface under various conditions, the mechanism of the conditioning process in this system is being studied. The actual contact between bubble and mineral in a flotation cell occurs so rapidly that the process cannot be recorded by high-speed photography. An apparatus has been devised which, in effect, retards contact so that some of the factors which govern the process may be studied in detail. From a knowledge of these factors it is intended to determine whether the process of contact can be accelerated in the flotation cell, thereby increasing the capacity of a flotation plant.

Where long paraffin-chain compounds are used as collectors it is difficult to produce the brittle unstable froths necessary for selective flotation. This is true, for instance, of the flotation of the important

ore minerals, scheelite (tungsten) and cassiterite (tin). It has been found that the difficulty arises from an interaction of the collector with the commonly used frothers. The nature of this interaction is being studied by measuring the surface tensions and surface viscosities of the solutions in relation to their frothing characteristics. A parallel approach to the problem of frothing is being made by measuring the persistence of single bubbles as a unit of froth.

#### Coal research section

An investigation into the gas-making, coke-making and by-product-making properties of Australian coals is being undertaken in co-operation with the National Gas Association, using equipment located in the Association's laboratories in Melbourne. To establish a basis for comparison, samples of the Greta Seam have been tested, and the Lithgow Seam is now being investigated.

Petrological investigations carried out during the year included a study of the spore content of coals from borehole samples in the Styx River area, Queensland, and an examination of thin sections prepared from a pillar sample of the Greta Seam, New South Wales.

At the Geology Department of the University of Sydney, fundamental petrological studies are to be made on a co-operative basis. These will include *inter alia*, a study of coke structure using x-ray techniques, an investigation of the occurrence and distribution of mineral matter in coal, and an investigation of the chemical variation and characteristics exhibited by vitrains and fusains throughout Australian coal-fields.

#### Gasification of brown coal

The Chemical Engineering Section's programme of fuel utilisation research has been considerably expanded. At present, work is centred on the investigation of the problems involved in the combustion and in the complete gasification of brown coal from the Latrobe Valley, Victoria. The complete gasification of brown coal is being investigated with two aims: (1) production of a gas suitable for the synthesis of liquid fuels and chemicals; (2) production of a gas of high calorific value suitable for distribution as a town gas. Possible methods of gasification were considered in relation to the properties of this coal and to developments overseas, and it was decided initially to investigate fluid-bed gasification procedures at elevated pressures for both purposes. The two small-scale plants now in the course of development will be of the minimum size to supply adequate data on the chemistry of the processes and the characteristics of the fuel beds, and to serve as a testing ground for the devices developed from study of the many associated problems, such as dust removal and gas cleaning, ash removal and coal feeding.

Investigation of fluid-bed gasification and fluid-bed carbonisation of brown coal, and studies of the kinetics of the gasification reaction of brown coal chars are also in progress.

#### Physical metallurgy

The Section of Physical Metallurgy continues to work in conjunction with the Research School of Metallurgy in the Baillieu Laboratory at the University of Melbourne, under the general direction of the Research Professor of Metallurgy.

The work on the strength and structure of metals, a collaborative project with the Aeronautics Laboratory of the Ministry of Supply (formerly the C.S.I.R.O. Division of Aeronautics) has continued, attention being paid to the mechanism of failure of metals by fatigue and to the influence of rate of strain on the mechanism of deformation at high temperatures.

The main work of the Section, however, has consisted of a study of the constitution and properties of the alloys of titanium, of the allotropy of titanium and of the influence of prolonged stress on the deformation of alloys of lead.

#### Titanium and its alloys

Some pure titanium prepared by the thermal reduction of the iodide was procured during the year to supplement the supply of 'Kroll' titanium donated by the United States of America Bureau of Mines.

The allotropic transformation at 882°C. was examined by the hydrogen-pressure method developed in the Section, by electrical resistance and by thermo-electric force determinations. The transformation was shown to be isothermal—a point on which some doubt had been thrown by earlier work—and the changes in properties are of sufficient magnitude to be used for examining phase relationships. In Kroll titanium, owing to the presence of impurities, the transformation is spread over a range of about 100°C.

The constitution of several alloy systems has been determined. Using pure titanium, the system hydrogen-titanium was examined up to concentrations of the order of 60 atomic per cent. hydrogen, as this system is utilised in the hydrogen-pressure method for examining other systems. Some thermodynamic relationships relating to the system were also examined.

The influence of iron, copper and chromium on the  $\alpha/\beta$  allotropic change has been studied as far as 5 atomic per cent. of each element. In each case there is a lowering of the allotropic change temperature. The hydrogen-pressure method was used. The solid solubility of these elements in the  $\alpha$  form of titanium has been shown to be very low in spite of a favourable atomic size factor. The solubilities are: Copper, less than 1 atomic per cent., and iron and chromium, less than 0.1 atomic per cent.

Using Kroll titanium (containing about

0.3% impurities), a constitution diagram for alloys containing up to 50 atomic per cent. iron has been worked out by microscopic and x-ray diffraction methods. The system is eutectiferous, the melting point of titanium being rapidly lowered by additions of iron. In the solid state the system shows a eutectoid change at about 600°C. The mechanical properties of alloys subjected to various heat treatments based on the diagram have received preliminary examination. It has been shown that a 4% iron alloy, after quenching followed by ageing treatment, can develop a tensile strength of the order of 100 tons/sq. in. with 4 to 5% elongation.

Also using Kroll titanium, preliminary work on the constitution of oxygen-titanium alloys has been carried out. It has been shown that oxygen raises the allotropic transformation temperature. Dissolving of oxygen considerably raises the hardness of titanium.

This latter fact has been used to create a hard surface layer by exposing titanium to oxygen at various temperatures and pressures. It has been shown that it is possible to increase the surface hardness up to 600 D.P.N. by increasing the oxygen concentration.

#### Creep of lead alloys

The creep characteristics of a very pure industrial lead have been examined under stresses of 150 to 500 lb./sq. in. The specimens were extruded in the laboratory, and in checking the absence of strain in the specimens after annealing it was found that a skin effect was present. This did not systematically affect the creep rate but gave a misleading idea of the annealed state. Removing 0.001 in. by etching was satisfactory.

Addition of tin to lead (0.01 to 0.9%) within the limits of solid solubility was made. Extruded and annealed specimens were stressed at 500 and 300 lb./sq. in. respectively. At the higher stress, the creep rate decreased with increasing tin content, but at the lower stress, tin concentration made no significant difference.

Additions of thallium to lead have also been made. This element has a much higher solid solubility range than tin. Alloys carrying between 0.5 and 40% thallium have been prepared, and test specimens made by extrusion and annealing. Both at 500 and 300 lb./sq. in. stress the creep rate is a complex function of composition and further work is in progress.

An alloy containing 2.5% thallium is being used to check the effect of strain rate on the mechanism of creep. Microscopic and x-ray back-reflection techniques have given results conforming with those obtained by other workers in the laboratory on aluminium.

#### Ceramics

To assist the ceramic industry, a survey and evaluation of some of its most im-

portant raw materials is being made in collaboration with the University of Melbourne and the Mines Department of several States. An extensive research aimed at improving the performance of refractories, particularly for the gas and cement industries, is well under way.

#### Minerals utilisation

The Minerals Utilisation Section was set up to investigate the properties of certain naturally occurring minerals. Work has been undertaken to improve the chemical processing of monazite to give high overall recovery of the constituents. High-temperature chlorination of monazite was explored as a means of producing phosphorous-free dry chlorides of the rare earth metals of the cerium group, with concurrent isolation of thorium chloride.

Studies on titanium compounds derived from the mineral rutile, via titanium tetrachloride, have been chiefly directed to

titanium nitride, and its use as the essential component of a hard metal.

Long-term studies on the separation and purification of the closely associated hafnium and zirconium by the lower anhydrous chloride method have been continued. Some preliminary tests were made on a modified process for preparing ductile zirconium metal.

Considerable progress has been made in developing a selective solvent process for extracting uranium compounds from low-grade titaniferous ores.

#### Chemical physics

A wide range of equipment is available for x-ray microscopy, mass spectroscopy, electron diffraction and electron microscope investigations, and many problems are being studied. A variety of equipment has also been designed and constructed by the mechanical, electrical and glass-working shops.

## An Introduction to Chemical Engineering

IT has always proved somewhat difficult to define the scope of chemical engineering both in the practical and in the theoretical fields. This book\* by Dr. Rumford is the first modern British text covering what are commonly called 'unit operations' and which form such an essential part of the subject. For those chemists who turn to chemical engineering and for that increasing body of students who take up the subject this will be found the most satisfactory introductory book at present available.

Dr. Rumford has met the difficult problem of condensing the wide range of material available by giving in each chapter a simple outline of the theoretical principles and then a description of the various types of industrial plants. The theoretical approach is introductory but quite adequate to enable the mode of operation of the various units to be understood, but naturally is neither sufficiently detailed nor extensive to be of use for design purposes. However, there are a number of neat expositions which reveal the author's long experience in teaching and examining in the subject.

The descriptive accounts are illustrated by a large number of half-sectional drawings which enable a clear picture to be obtained of the general construction of the equipment, but they would have been improved by the use of a bolder outline, together with some indication of the range of sizes of the units. At the same time some very useful figures of the capacity of the plants are given which will be of particular value to the student. The

descriptive sections are really confined to the physical aspects and no attempt is made to give any engineering details of construction.

The various chapters cover the operations of heat transfer, distillation, crystallisation, filtration, etc., but there is no specific chapter on fluid flow, although this is partly offset by the first chapter entitled 'Chemical Works Pumping.' This is, however, largely descriptive of the types of pumps used and the theoretical aspects are treated rather too sketchily for so important an operation. There is a welcome chapter on automatic control and one on gas cleaning, but it is unfortunate that the author has not been able to include a chapter on the various types of reaction vessels which form the heart of so much of the chemical process industries.

The limitation in the theoretical treatment can be seen in the chapter on absorption where no consideration is given to the basic importance of the fundamentals of diffusion, no detailed examination of the factors affecting transfer coefficients and no reference to the recent work on flooding velocities in columns. Again, the chapter on heat transfer does not give a good idea of the limitations in the application of the equations to practical problems.

The book is valuable in that it provides a readily understandable picture of the forms of chemical plants, whilst at the same time giving a simplified account of the principles on which they are based. The general style of writing makes the book easy to read, though parts of the theoretical treatment are too compressed for easy understanding and many would like the nomenclature and units to be more precisely defined.

J. M. COULSON, M.A., Ph.D

\**Chemical Engineering Operations: An Introduction to the Study of Chemical Plant*, by F. Rumford. Constable, London, 1951. Pp. 376 in index, illustrated, 30s.



# New Plant and Equipment

## Catalytic gas purifier

Many industrial processes require gases which are virtually free from oxygen. This is particularly desirable in certain metallurgical operations such as bright annealing, or the manufacture of powder metals and in chemical operations such as the synthesis of ammonia. Commercial hydrogen or nitrogen contain some oxygen up to 0.3% or even higher, and the presence of even this small amount of oxygen can cause unexpected difficulties. A new method of removing this oxygen has been introduced by Baker Platinum Ltd., who claim that after simple passage of the gas through their purifier at room temperature, less than 1 p.p.m. of oxygen remains.

In the Baker *Deoxo* system the removal of oxygen from hydrogen or from inert gases is accomplished by passing the gas over a specially prepared catalyst, which brings about combination of hydrogen and oxygen in the ratio of 2:1 by volume to form water vapour. This reaction will take place at room temperature. The resultant water vapour is carried away in the gas stream and can be removed by any standard dehydrator.

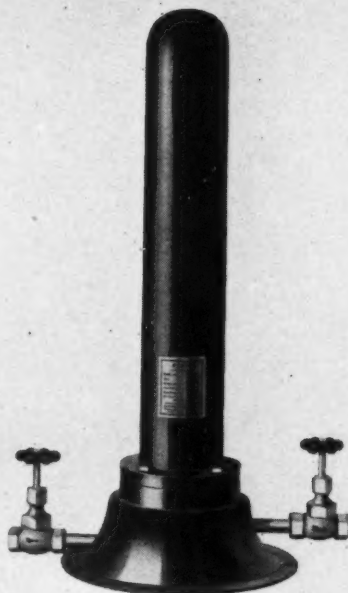
The heat generated by the catalytic reaction amounts to a temperature rise of approximately 10°C. for each 0.1% of oxygen removed. The standard equipment will operate at any pressure up to 50 lb./sq. in.

The Baker *Deoxo* units are of simple, compact and rugged construction. They involve no operating or maintenance expenses and no electrical current, auxiliary heating or water cooling are required. The unit is connected to the gas supply line and, it is claimed, it will operate indefinitely without attention, provided that the installation instructions are followed.

From the nature of the reaction it will be clear that unwanted oxygen or hydrogen can be removed from inert gases such as nitrogen, argon, helium, neon, carbon dioxide, and saturated hydrocarbons, provided that there is always a ratio of two volumes hydrogen to one volume oxygen in the gases to be treated. If one or other of these gases is not present in the free state, it must be added to the gas stream ahead of the purifier in the correct volume necessary to effect a complete combination.

It is proposed to offer the units in the following range of sizes: 5, 200, 1,000, 2,500 and 5,000 cu. ft./hr. capacities.

The firm's Research Department is now developing units to provide for the catalytic combination of: (a) oxygen and hydrogen in the presence of carbon monoxide, (b) oxygen and carbon monoxide to form carbon dioxide in gases that do not contain hydrogen, (c) carbon monoxide in hydrogen to form carbon dioxide, and (d) carbon monoxide and hydrogen to form methane and water vapour.



Unit for catalytic removal of oxygen from industrial gases.

## High-pressure piston valve

Primarily designed for instrument work, a new British valve is claimed to permit controlled flow to an extent hitherto unknown. Tested hydraulically to 9,000 lb./sq.in., this  $\frac{3}{16}$  in. bore high-pressure valve is suitable for oil working pressures up to 5,000 lb./sq.in. and for use with superheated steam up to 2,000 lb./sq.in. Added life and strength is achieved by having the diameter of the spindle larger than that of the piston, so that the load exerted on the thread when operating the valve is very small. Throttling is said to have no adverse effect on the efficiency of the valve, as wire-drawing will be ex-



Piston valve suitable for pressures up to 5,000 lb./sq.in.

perienced only on the bottom of the piston, whereas the sides of the piston, which actually seal the valve, remain unaffected. Maintenance is straightforward and when, after long service, wear takes place, resulting leakages can be corrected immediately by tightening the bonnet nut, thus compressing the two valve rings more closely around the piston.

The body is a high-quality steel drop forging with flats 1 in. apart. The piston, bonnet nut, lantern bush and spindle are of stainless steel and the bonnet is carbon steel. The valve is manufactured by Richard Klinger Ltd.

## Industrial gasholder

Although the Wiggins gasholder design dates back more than 15 years, when the principle of confining gases and vapour by means of a synthetic rubber coated fabric was first conceived, the application of this type of gasholder for storage of chemical process and industrial gases at higher pressures was only initiated about three years ago. Today, installations ranging up to 500,000 cu. ft. are being used for storing carbon dioxide, carbon monoxide, nitrogen, hydrogen, natural gas, sewage gas, oxygen, tetrafluoroethylene, and coke oven gas, and gasholders up to 7,000,000 cu. ft. capacity are now being considered.

The Wiggins gasholder, marketed by Ashmore, Benson, Pease & Co., Ltd., is essentially a piston displacement type gasholder as opposed to a bell or lift type water gasholder. The seal medium used to prevent escape of gas through the annular space between piston and shell is a flexible impermeable synthetic rubber coated fabric. The seals are fabric cylinders whose edges are joined by means of gas-tight closures to the piston telescoping fender and the shell wall. The seal fabric in general use for most gasholders today consists of a core of asbestos cloth that is impregnated and coated with a Neoprene rubber coating. The asbestos cloth used is so designed to give the strength required with a weave that will permit the coating to key through the cloth and give maximum peel strength. Neoprene coating is used because of its excellent resistance to ageing, permeability and chemical reaction.

The advantages of this design of gasholder are claimed to be (1) water has no effect on its operation, (2) mechanically, the holder is simplified by the elimination of close tolerances, (3) routine servicing has been eliminated by providing the balancing system with self-lubricating bearings and non-corrosive parts, (4) gas composition is unchanged and it is possible to store dry gases and keep them dry, (5) safety of operation and insurance against gas loss are provided by the fabric seal, (6) operating pressure can be designed to meet any particular specification up to 20 in. of water and (7) foundation requirements are reduced because the weight of the Wiggins structure is less and foundation settling is no hazard to continued operation.

# World News

## GREAT BRITAIN

### Carbon black factory opened

The Philblack Ltd. carbon black factory at Avonmouth, Bristol, was officially opened last month, when over 200 representatives of the rubber and other consumer industries visited the plant. There are two units. The 'O' unit, which started operation in April 1951, has a capacity in excess of 30,000,000 lb. p.a. of Philblack O, a high-abrasive black for tyre treads and belting. The 'A' unit, which started early in July, has a capacity of 20,000,000 lb. p.a. of Philblack A, a medium reinforcing black for tyre carcasses, cable sheathing, rubber boots, etc. From the first day of operation both units have been producing the oil-blacks on a commercial basis. A description of the plant was given in INTERNATIONAL CHEMICAL ENGINEERING, December 1950.

### Chemical manufacturers' dinner

The need for increased productivity in the chemical industry to overcome shortages of raw materials was stressed by Mr. C. G. Hayman, chairman, at the annual dinner of the Association of British Chemical Manufacturers in London last month. This plea was reinforced by the chief guest, Mr. William Batt, chief of the E.C.A. mission to the United Kingdom, who said that raw material shortages were likely to remain with us because 'It is amazing what an all-out effort with full employment can do in the way of chewing up raw materials.'

More than 800 attended the dinner, a record gathering. Among the guests were Sir Harold Hartley, president of the Institution of Chemical Engineers.

### Powell Duffryn's new interests

Developments in the chemical engineering field made by Powell Duffryn Ltd. were surveyed by Sir Herbert Merrett, chairman of the company, at the annual general meeting.

Production at the Hayes factory of Powell Duffryn Carbon Products Ltd. is proceeding and sales are said to be satisfactory. The laboratories which were formerly at Battersea have now been removed to Hayes. Manufacture and trading in this field have recently been extended by the formation of a company in U.S.A. jointly with the Great Lakes Carbon Corp., known as the British American Carbon Corpn.

Powell Duffryn Ltd. have a 50% partnership with Socony-Vacuum Co. Inc., New York, in the British company, Vacuum Oil Co. Ltd. The construction of the firm's oil-refinery, which started a year ago, has proceeded almost to schedule. Amplifications have been made in the planned layout, the chief one being the

incorporation of a catalytic cracking unit which will allow the refinery to improve the quality and increase the quantity of petrol produced. Through its association with the American company Vacuum Oil Co. enjoys the benefit of the latest information in oil-refining research and technique and this source of knowledge was used in deciding on the installation of the catalytic cracking unit.

In other directions, oil business is expanding through a subsidiary, Cory Bros. & Co. Ltd., and storage and bunkering facilities at Las Palmas have been enlarged to deal with a throughput of up to 1,000,000 tons p.a. Facilities have also been increased at Barry Dock by more than 30,000 tons and a further tankage of 30,000 tons is also being added, which will bring the company's storage at that port up to more than 120,000 tons. Through a subsidiary, Millers & Corys, the turnover at the oil-bunkering depot at St. Vincent, Cape Verde Islands, is steadily increasing. During the past year a new oil depot has been opened at La Goulette through associates in Tunisia.

### Nickel and cobalt allocations

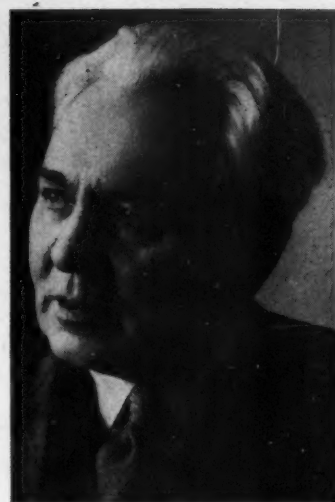
For the fourth quarter of 1951 the Manganese-Nickel-Cobalt Committee of the International Materials Conference have recommended the allocation of 5,088 tons of nickel and 329.1 tons of cobalt to the United Kingdom. The United States is to receive 19,690 tons of nickel and 1,212.4 tons of cobalt. The 32 governments represented on the committee have accepted its recommendations. Total nickel production for this period is expected to be 31,500 metric tons, while requirements amount to 56,800 metric tons, a deficit of 25,300. Cobalt production is estimated at 2,075 tons, while requirements amount to over 4,000 tons, a deficit of about 2,000 tons.

### Cheaper lead

According to a Ministry of Materials announcement, the price of imported good soft lead has been reduced from £180 to £175 per ton delivered consumers' works. This reduction is made possible by a new agreement with Commonwealth producers. Following this, the Ministry of Supply has made a new order reducing the price of remelted lead and lead scrap by £5 per ton. The new prices per ton are remelted lead £160, scrap cable sheathing £160, and lead scrap other than cable sheathing £156.

### Sulphuric acid dearer

The Minister of Materials has made an Order permitting increases on the maximum prices of sulphuric acid. These increases are due to the rise in the price of imported pyrites and in freight rates for both sulphur and pyrites. The additions



Dr. N. E. RAMBUSH, M.I.Chem.E.

who has been elected chairman of the Power-Gas Corporation Ltd. He joined the company in 1918 as chief engineer and was appointed managing director of the company and its subsidiaries in 1937. Since 1947, he has been vice-chairman. His book, 'Modern Gas Producers,' published in 1923, is still recognised as one of the major works on the subject.

to maximum prices are 14s. per ton on weak acid (77%  $H_2SO_4$ ) and 27s. per ton on strong acid (more than 84.02%  $H_2SO_4$ ).

### Copper export restrictions

For the two quarters since April 1, 1951, exports of semi-manufactures of copper and copper alloys have been restricted to approximately half the rate prevailing in the first six months of 1950. The Board of Trade now announce that, because of the continuing world shortage in the supply of the raw materials involved, these restrictions are to be extended in the same form for a further three months until the end of the year. The export of semi-manufactures of zinc will continue to be permitted only in exceptional circumstances. The validity of existing licences is not affected by this Order.

### Steelwork productivity

A constructional steelwork productivity team has visited the U.S.A. to study American steelwork practice. In common with other such teams sent under the auspices of the Anglo-American Council on Productivity, the 14 members were made up in equal proportions from the various levels of industry, i.e. management, technicians and operatives. Some of the team were nominated by chemical engineering firms, which included Ashmore, Benson, Pease & Co. Ltd., Babcock & Wilcox Ltd., Head Wrightson & Co. Ltd., International Combustion Ltd., and Newton Chambers & Co. Ltd.

Visits were arranged to American firms fabricating structural steelwork, bridges, oil tanks, and accessories, as well as to an ore storage and handling installation.



### Bigger styrene plant

The plant being constructed at Grange-mouth by Forth Chemicals Ltd. for the production of monomeric styrene is to be expanded and output will be substantially increased. The smaller plant is expected to come into production next year, and the extensions about two years later.

Forth Chemicals Ltd. was formed last year by British Petroleum Chemicals Ltd. (jointly owned by Anglo-Iranian Oil Co. Ltd. and The Distillers Co. Ltd.) and Monsanto Chemicals Ltd. One of the raw materials of monomeric styrene is ethylene, which will be supplied from the new cracking plant which has recently been commissioned by British Petroleum Chemicals Ltd.

### Sulphur prospecting project

A Sulphur Exploration Syndicate composed of the principal consumers of sulphur in the U.K. has been formed to explore sources of sulphur all over the world which might be eventually exploited. A limited company, The British Sulphur Corp. Ltd., has now been registered with a nominal capital under the auspices of the syndicate. The purpose of the registration of this company is to provide a vehicle for the exploitation, if required, of properties considered of interest by the syndicate. The eventual form of the company, its final directorate, and finance, are all matters which will be decided in the light of future requirements. The members of the syndicate will not necessarily participate financially in the activities of the company, and the amount of finance required and its source are matters on which no decision has been taken.

### Mass production engineering

To study methods of inspection in practice in American plants engaged in mass production, mainly engineering, a team of eight specialists visited the U.S.A. last month. In the U.S. they hope to examine inspection methods applicable to raw materials, semi-raw materials and finished articles.

Primarily, attention will be focused on the function exercised (or exercisable) by inspection in the organisation of industry at large. Such matters as the status of inspection departments in relation to other departments, the bearing of inspection on testing, the application of automatic jigs and fixtures, and the development of electronic tests are also expected to be discussed by the team in the report to be issued later.

Mr. Reginald F. Norris has relinquished his position with the oil engine division of the Brush Aboe Group to become general manager of D.M.M. (Machinery) Ltd., who are the English representatives of a group of German engineering firms, including Carl Canzler, the chemical plant manufacturers.

### New chairman of I.C.I. Plastics

Mr. J. C. Swallow has been appointed chairman of I.C.I. Plastics Division in succession to Mr. P. C. Allen, who becomes a member of the I.C.I. Board and Group Director responsible for the Plastics, Paints, and Leathercloth Divisions. The new chairman was formerly joint managing director. He has been associated with I.C.I. since its formation and throughout his scientific career has taken a very active part in plastics research. This year Mr. Swallow delivered a series of Cantor Lectures to the Royal Society of Arts on 'The Plastics Industry.'

Dr. J. E. Sisson has been appointed joint managing director of the I.C.I. Plastics Division. He joined I.C.I. in 1933.

### New chairman of I.C.I., Billingham

Dr. G. I. Higson has been appointed chairman of the Billingham Division of I.C.I. He joined the Billingham laboratories and works in 1922. Dr. R. Holroyd has been appointed joint managing director in his place. He joined I.C.I. in 1928.

### New British standards

**Hexagon bolts.** The publication of two standards (B.S. 1768:1951 and B.S. 1769:1951) for unified hexagon bolts, screws and nuts marks the most important development to date in the work of standardisation between the three North Atlantic Treaty countries since the signing of the 1948 'Declaration of Accord.' It will now make possible really effective implementation of the agreement to adopt the unified screw thread, the standard for which was published as B.S. 1580 in 1949.

At a conference between industrial and Service delegates from the U.K., Canada and the U.S.A., held at the end of April under the aegis of the British Standards Institution, agreement was reached in principle on the basic dimensions of hexagon bolts, screws and nuts, and overall limits were established for two series, normal and heavy series, based essentially on a selection from existing American standards.

Apart from the military significance of this development, it will lead to interchangeability of many products in a wide range of engineering industries.

**Portable fire extinguishers.** A further document in the series of standards covering portable fire extinguishers, viz. B.S. 1721, 'Portable fire extinguishers of the carbon tetrachloride type,' has been published. This standard is an entire revision of that part of B.S. 740 which covered carbon tetrachloride fire extinguishers and all types of carbon tetrachloride fire extinguishers are now provided for. It formulates details of the materials to be used, the type and dimensions of the various parts, as well as details of construction and gives methods of testing the fire extinguishers at various stages of manufacture, and provides details of marking.

Copies of these standards may be ob-

tained from the British Standards Institution, 24 Victoria Street, London, S.W.1, prices, B.S. 1768, 3s.; B.S. 1769, 2s. 6d.; B.S. 1721, 4s., post free.

### Directory of chemical consultants

The second edition of the 'Directory of Independent Consultants in Chemistry and Related Subjects' has now been published by the Royal Institute of Chemistry. Like the first edition issued in 1946 this publication contains particulars of independent consulting practices, concerned with any aspects of chemistry in which fellows or associates of the Institute are principals. The general character of the directory is unchanged, but the separate geographical index has been omitted and the relevant information incorporated in the subject index, which has been made more detailed in this and other respects in order to facilitate reference.

Copies of the new edition of the directory have been forwarded to the consultants mentioned in it and to various organisations and individuals known to be interested. Copies will be sent, free of charge, to all who enquire about consulting services.

### Purchasing Officers Association

At the recent conference of the Purchasing Officers Association in Llandudno, Mr. T. L. Garner, Precision Rubbers Ltd., was elected president for the year 1951-2. Two of the six vice-presidents are J. C. Thompson, Edgar Allen & Co. Ltd., and C. H. Tomlinson, Boots Pure Drug Co. Ltd. The new chairman is Mr. H. W. King, Eburite Containers Ltd., the vice-chairman, Mr. F. J. White, Chance Bros. Ltd., and the honorary treasurer, Mr. R. J. Mitchell, The Morgan Crucible Co. Ltd.

## GERMANY

### Synthetic rubber industry

Production of butadiene at the Hüls plant was scheduled to begin last month, and of synthetic rubber this month. From December, the synthetic rubber should be on the market, though initially not more than 500 or 600 tons a month as only one production line in the plant is workable, and the only other plant in Western Germany has been completely dismantled.

Earlier the U.S. Senate Armed Services Sub-Committee calculated that an annual rate of 7,000 tons could be reached within 3 or 4 months, and possibly 35,000 tons after 18 months of rehabilitation. It pointed out that Germany had a peak capacity of 74,000 tons a year during the war.

Germany's biggest pre-war and war-time producer was the plant at Schkopau, which is in the Russian zone. West German estimates of Schkopau's present output range 2,000 to 4,000 tons a month from a capacity of perhaps 5,000 a month.

Of the initial Hüls production, about 100 tons a month is expected to be per-

bunan (the original Buna-N which is equivalent to the American synthetic GR-N), the oil-resisting synthetic. It is calculated that with adequate priorities for capital and materials, the Hüls plant could produce about 1,500 tons a month by about the end of 1952. This could include about 500 tons of perbunan, enough to supply Europe's requirements of that specialised product.

Output of all synthetic could reach 4,000 tons a month within about 3 or 4 years. This would supply about half of Germany's total present consumption of rubber, which is about 90,000 tons a year. German rubber manufacturers assert that production of some synthetic rubber is indispensable. On strictly functional grounds the desired proportion is put at about 20% of total rubber consumption, including about 2% of perbunan. While American synthetic rubber is only about half the present price of natural rubber, they would like to use a much larger proportion of it.

The unknown factor is how the Hüls rubber will compete with the American synthetic that Germany at present imports. Some industrial users doubt whether the qualities produced will be as usable as the American types, but the general impression is that the German rubbers will be as good as the American, or will be made so.

There is rather less confidence that the production costs at Hüls, when in quantity production, will be competitive with the American. As regards the question of whether it will be worth while to expand Hüls output merely to save dollars, some Allied financial experts contend that, in the near future, foreign exchange is likely to be one of the few things that are not scarce in Germany. If they are right, dollar-saving will be a secondary consideration.

Export demand for German rubber goods is stated to be relatively good, as they are competitive in price and delivery dates. But home business, as in many other lines, has lately been poor, mainly because the public is short of money.

#### Co-management refused

The Allied High Commission has refused to make a law providing for workers' co-management of West German chemical industries, according to a report from Bonn. The matter is up to the West German Government and Parliament, the Commission is alleged to have stated.

After controversy throughout the country, Parliament last spring passed a law giving coal, iron and steel workers a half share in the management of their firms. The chemical industries are next on the trade unions' list for co-management.

#### International chemical congress

The French organisation, Société de Chimie Industrielle, will hold the 25th International Congress of Industrial Chemis-

try at Frankfurt-am-Main from May 18-25, 1952. The occasion will coincide with the Achema, the exhibition of chemical equipment organised by the Deutsche Gesellschaft für chemisches Apparatewesen. Other chemical, engineering and metallurgical institutions will be holding meetings in Frankfurt at the same time.

### FRANCE

#### Polythene plant planned

Distribution of polythene in France will henceforth be under the control of the Chemical Board of the French Ministry of Industry and Commerce. This decision was taken owing to the scarcity of this product, supplies of which come mainly from Britain.

To meet increased demand in France, a French company, Ethylene-plastique, was set up on May 31 last in Douai (northern France) to produce polythene under the licence of Imperial Chemical Industries Ltd. Construction of a factory in Mazingarbe (Pas-de-Calais, northern France), near the ethylene-producing areas, will start soon.

The new factory is expected to start production by the end of 1953. Initial output capacity will be 2,000 tons per year. This figure, however, was considered to be too low to meet home requirements and might be quickly increased.

#### Paper from tropical timber

A recent French process may ease France's paper shortage. A State-sponsored undertaking, Régie Industrielle de la Cellulose Coloniale, has developed a process which makes it possible to manufacture paper pulp from short-stapled African tropical timber. Until now only soft, resinous long staple timbers could be used for this purpose.

A pilot plant has started operation at Bibresso, near Abadjan (Ivory Coast). Its initial output is 6,000 tons of paper pulp a year, which will be increased to 10,000 tons later. Experience has shown that the new process is commercially profitable and the paper as good as that produced from the usual material. Its price will not exceed that of imported paper.

The company is now negotiating with private firms for capital for the erection of a big plant in Cameroon to produce 30,000 tons of pulp p.a. Its production costs are expected to be 15 to 18% lower than present Bibresso prices. The plant could go into operation within 12 to 18 months. If the necessary funds can be raised other plants may be set up.

The resulting expansion in the consumption of timber would be of considerable importance to French Colonial Africa. At the moment only a small proportion of finer woods, such as mahogany, can be profitably marketed.

#### Synthetic rubber from alcohol

An organisation known as the Société d'Etudes du Caoutchouc Synthétique à base d'Alcool has been formed in France at the instigation of the Institut Français de l'Alcool and the Confédération Générale des Planteurs de Betteraves. The new organisation, which has a capital of 12,500,000 French francs, will study all aspects of the establishment and operation of factories for the production of synthetic rubber and its by-products from alcohol.

It is tentatively planned to use some 12,000,000 hectolitres of alcohol for the manufacture of 35,000 to 40,000 tons of GRS rubber.

#### Manufacture of artificial gems

The manufacture of artificial gems has recently been started on a commercial scale in the Doubs area of France, where some five factories are producing artificial rubies. Indigenous white bauxite ores are used and the operation is carried out in an electric furnace at a high temperature, the bauxite being mixed with very pure carbon. A sixth factory is being built at Charquemont and, when in operation, production will be sufficient to permit export. Production of artificial corundum, used in particular in the abrasives industry, is being carried on at Beyrède.

Previously French bauxite had been exported to Czechoslovakia, where extensive production of synthetic gems for industrial and ornamental purposes was carried on.

### ITALY

#### Magnesite output to be raised

The Societa Magnesite Valdelsa, which operates Italy's largest active magnesite mine, plans to increase its output to some 3,000 to 4,000 tons annually, which would cover almost one-third of Italy's total requirements. Imports of natural magnesite in 1950, most of which came from Greece, exceeded by almost 60% those of 1949. Imports of calcined or caustic magnesia, nearly all of which came from Austria, were 40% higher.

### BELGIUM

#### New chemical company

A new 10,000,000 franc company, Atlantic Chemicals, has been registered in Antwerp. The Atlantic Refining Co., Philadelphia, is among the shareholders.

### BELGIAN CONGO

#### Wood-pulp syndicate formed

A new syndicate has been formed to study the possibilities of wood-pulp manufacture in the Belgian Congo. The organisations participating in the syndicate are the Congo company Agrifor, the Government of the Belgian Congo, the U.S. Brown Paper Co. and the *Readers Digest*.





**NEW 'CAT CRACKER' FOR SHELL AT PERNIS, HOLLAND**

The catalytic cracking unit which was recently completed at Shell's Pernis refinery, near Rotterdam. From left to right can be seen the stack of the furnace (height 196 ft.), catalyst hoppers and, at the top, the conveyor for the catalyst. In the front is the compressor house. In the right background is the fractionating column. This is the first modern-type catalytic cracking unit to be constructed in Europe. After taking nearly two years to build, it is due to come into full operation shortly with a capacity of about 1,250,000 tons p.a. With this plant, a large part of the post-war expansion programme at Pernis has been completed. Some £20,000,000 has been spent since 1945, the catalytic cracker taking a substantial part. The total area of the Pernis refinery is 900 acres, and the storage capacity amounts to 1,200,000 tons. The refinery's daily consumption of fuel gas is 450,000 cu. m.; fuel oil, 500 tons; steam, 4,500 tons; and electricity, 200,000 kWh.

This is the second syndicate to be formed to study the possibilities of wood-pulp manufacture in the Belgian Congo. Another syndicate, formed last year, is concentrating its attention on the papyrus sedge which clusters thickly round the banks of the Congo river in the Katango, near its source.

#### INDIA

##### **Pyrites deposits discovered**

India may soon become a leading sulphur producer following the reported discovery of extensive deposits of pyrites in the southern hill tracts of Bombay State, which extend over 10 miles. Initial exploration and investigation had shown that an entire range of hills contains pyrites, which may yield several million tons of sulphur. When operations commence in the near future about 10,000 tons of pyrites will be cut monthly, with an estimated sulphur yield of 4,000 tons. It is expected that the entire internal need, estimated at 50,000 tons, could be met.

##### **Oil refinery project examined**

The Government of India have made a proposal to the three principal oil companies operating in India for setting up a 2,000,000-ton oil refinery in Bombay State or two 1,000,000-ton refineries in Bombay and Madras States. They have guaranteed immunity against nationalisation for at least 30 years and payment of due com-

pensation thereafter were nationalisation at all decided. A similar suggestion made in 1948 was turned down by the companies on account of the 'exceedingly heavy cost involved in the project.'

The Government of India is reported to have recently renewed the suggestion, in view of the oil developments in the Middle East, and asked them to reconsider their decision. The companies that would be agreeable to setting up the refinery plant would be required to make their own arrangements to import crude oil, preferably from sterling areas.

Under the proposal, the party working this scheme will be permitted to have full control over both capital and management and will be allowed to import all the crude oil necessary and to distribute the finished products with the exception of a very small fraction of the production.

The annual requirements of oil for the country are estimated at 3,000,000 tons. The only refinery at present working in India is at Digboi in Assam, which refines locally mined crude oil and meets a very small fraction of India's requirements.

##### **Synthetic petrol scheme**

The broad principles of a scheme submitted by the Koppers Co., U.S.A., to set up a synthetic petrol plant in India have been accepted by the Government. Koppers will set up two synthetic petrol plants. One will produce 70,000 tons of

aviation petrol, 28,000 tons of motor petrol, 1,800 tons of refined phenol (carbolic acid) and 500,000 tons of domestic coke a year, and a bigger plant will produce 100,000 tons of aviation petrol, 85,000 tons of motor petrol, 3,600 tons of refined phenol, and 1,000,000 tons of domestic coke.

##### **Synthetic oil project**

A private firm in Orissa has entered into an agreement with a German and some French firms for the production of synthetic oil. The foreign firms will be entrusted with the task of setting up a gas producer and undertaking construction work. The capital is being raised by the Indian firm partly in India and by a loan from a French bank.

#### YUGOSLAVIA

##### **Tannin from acorns**

A Croatian tanning extract factory has begun producing tannin from acorns, instead of from oak wood as hitherto. The acorn-tannin is said to be of the same quality as that produced from the wood. This new development will release some 8,000 tons of Yugoslavian oak annually for other purposes.

##### **Improved asbestos extraction**

The Belgrade Scientific Research Institute has developed a new chemical process for the extraction of asbestos from its ores. The new method replaces the grinding process employed hitherto and is being used for the first time on a commercial scale at the asbestos factory at Mladenovac in Serbia. It is expected that Yugoslavia will no longer require to import asbestos yarn as a result of the discovery.

#### BRAZIL

##### **Synthetic resins project**

Agreement has been reached between Reichhold Chemicals Inc., New York, and the Brazilian firm, Resana SA Industries, whereby the Brazilian firm will manufacture a complete line of the American firm's synthetic resins for the varnish, plywood, paper and textile industries, it is announced here. The plant will be set up in the vicinity of Sao Paulo.

##### **Polyvinyl factory planned**

Arrangements have been concluded between the U.S. Goodrich Chemical Co. and the S.A. Industrias Reunidas F. Matarazzo for the formation of a jointly-owned plastics manufacturing company, Geon do Brasil, with plants near Sao Paulo. The new plant will have an initial production capacity of 6,000,000 lb. of Geon polyvinyl materials a year.

Mr. S. W. Richardson, president of Goodrich, said that the plants will manufacture vinyl chloride monomer, Geon polyvinyl chloride resins and Geon plastic compounds. Engineering work is well

under way and it is estimated that the facilities will be completed in late 1952 or early 1953.

S. A. Industrias Reunidas F. Matarazzo operates 300 concerns and is one of the largest industrial firms in S. America. Goodrich is also associated with British Geon Ltd. and is currently constructing facilities in Japan through Japanese Geon Ltd., to produce Geon polyvinyl plastics (see INTERNATIONAL CHEMICAL ENGINEERING, March 1951, p. 137).

#### U.S.A.

##### New refinery catalysts plant

A new \$3,000,000 plant to manufacture synthetic cracking catalysts for the petroleum industry, has recently started production. It is owned by American Cyanamid Co. and substantially increases this company's output of catalyst.

The catalyst, consisting of tiny spheres about 0.002 in. in diameter, looks like a fine powder and flows like a liquid inside the cat cracking units. The spheres are so fine and porous that 1 lb. of the material offers an active surface of about 55 acres on which the cracking of petroleum takes place.

First of its kind built since 1942, the plant is a 22,000 sq. ft. structure occupying part of Cyanamid's 90-acre site near Michigan city. Part of the installation is a spray-drier, believed to be the largest of its kind in the world. The drier forms the catalyst into tiny spheres in much the same way as old shot towers formed molten lead into buckshot. In the spray-drier, however, the catalyst is atomised in a strong current of warm air, rather than by being dropped from the top of a tower as in the case of buckshot. Development of this microspheroidal catalyst was accomplished in co-operation with the petroleum industry.

##### Esso refinery expansion

The Esso Standard Oil Co. plans to spend more than \$35,000,000 for new construction at its Baton Rouge Refinery, according to Mr. H. Voorhies, general manager of the refinery. The project would expand the refinery's processing capacity from 240,000 to 270,000 barrels of crude oil daily in addition to a major expansion of its chemical operations. Construction would begin immediately and was expected to take about 2 years.

Expanded facilities would include new distillation equipment for crude oil and other petroleum fractions; expansion of fluid catalytic cracking facilities; additional equipment for processing petroleum gases to provide some of the raw materials for chemicals and materials for aviation petrol; expanded blending and shipping facilities for petrol, kerosine and heating oils. Facilities for producing additional butadiene, raw material for synthetic rubber, and for increasing the refinery's output of synthetic alcohols from petroleum are also scheduled as well as equipment for the production of benzene.

##### New plant will raise iron output

A \$75,000,000 plant for the processing of magnetic taconite ore at Beaver Bay, Minnesota, is to be erected by the Reserve Mining Co., Cleveland. The project is scheduled for completion in 4 years. Said to be the first to produce blast furnace feed in commercial tonnages from taconite ore, the plant will tap the vast resources of taconite in the Mesabi ranges, said to amount to nearly 1,500,000,000 tons. It will have a capacity to produce 2,500,000 tons of high grade iron ore from about treble that amount of taconite. The taconite will be pulverised into fine powder and iron mineral separated by a magnetic process from sandy waste material. Finally, the iron mineral will be rolled into small pellets and baked in a furnace. By this process, it is claimed, it will be possible to produce, at a competitive cost, iron ore pellets containing about 64% iron, from taconite with an iron content of as low as 25%.

##### Electronic process control

A new electronic scanning system, which can be used in chemical, petroleum and other processing industries in the production of synthetic fibres and yarns and in steam generating stations has been developed by the Minneapolis-Honeywell Regulator Co. Capable of controlling as much as 270 processing temperatures, the system records only those temperatures that deviate beyond a pre-set limit. It includes an alarm that allows for immediate correction of excess temperatures.

##### Powder metallurgy symposium

A symposium on testing metal powders and metal powder products, consisting of several papers by leading authorities in this field, will be the technical feature of the 1952 Spring Meeting of the American Society for Testing Materials to be held during the week of March 3 at the Hotel Statler in Cleveland, Ohio. While many symposia have been held on the scientific and technical aspects of powder metallurgy, this is believed to be the first which is specifically concerned with methods of testing and should serve as a medium of exchange of information for all who are interested in this field. Papers which are concerned with any phase of the testing of metal powders or of parts fabricated are invited.

##### A.S.T.M. anniversary

During the week of June 23, 1952, the 50th anniversary meeting of the American Society for Testing Materials is to be held in New York City. Numerous special symposia are being developed as part of the technical programme and it is expected they will include the following subjects: synthetic fibres, adhesives, continuous analysis of water, plastics, conditioning and weathering, light microscopy, fatigue, effect of temperature, and tin. In all, some 35 sessions will be held.

#### SOUTH AFRICA

##### Rayon factory to be built

A £7,500,000 rayon plant is to be built on a site at Umkomaas, Natal. The project, sponsored by overseas investors and supported by South Africa's Industrial Development Corporation, originated over a year ago; it was found that the saligna gum trees in Zululand were suitable for rayon production.

It is understood that options have already been exercised for the purchase of thousands of acres of forest in the Kwanbonambi area of Zululand.

#### CANADA

##### Formaldehyde plant scheme

Pentaerythritol may be produced for the first time in Canada in a \$2,000,000 chemical plant planned to be built in Quebec and financed jointly by the Shawinigan Chemicals Ltd., Montreal, and the Heyden Chemicals Ltd., New York, it is learned here. If built, the plant is expected to have an annual capacity of 30,000,000 lb. of formaldehyde and 3,000,000 lb. of pentaerythritol. It is also planned to produce other chemical products there at a later date.

**Stainless steel products.** The recently published official directory of the Stainless Steel Fabricators' Association of Great Britain contains details of the stainless steel products fabricated by its 47 members. An alphabetical list of members is followed by a classified list of products indicating the respective manufacturers. Ninety-six pages are devoted to members' announcements which give comprehensive illustrated notes on the wide range of products manufactured for domestic and industrial purposes. The stainless steel chemical equipment detailed includes reaction vessels, condensers, crystallisers, mixers, insulated storage tanks, vats, boiling pans, pressure vessels, heat exchangers, solvent-recovery plant, petroleum equipment, sterilisation and pasteurisation equipment for milk, beer, wine, etc., and a vacuum-filling and capping machine. At the beginning of the book several pages are devoted to the history of stainless steel. This traces the development of modern steels from the discovery of lead chromate in Siberia in 1766. The directory is called 'British-Made Products in Stainless Steel' (pp. 136, price 10s. 6d.).

##### Contributions

THE EDITOR welcomes practical articles and notes on chemical engineering and industrial chemical subjects with a view to publication. All contributions, which should be fully illustrated whenever possible, are carefully considered. A preliminary letter or synopsis is advisable. Address such material to THE EDITOR, INTERNATIONAL CHEMICAL ENGINEERING, 17 Stratford Place, London, W.1.



# MEETINGS

## Institution of Chemical Engineers

November 10. 'The Field of Rubber and Allied Products in Chemical Engineering,' by S. A. Brazier, 3 p.m., Reynolds Hall, College of Technology, Manchester.

December 1. 'Study of Carbide Furnace Operation,' by R. B. Peacock, 2.30 p.m., Radiant House, Bold Street, Liverpool.

December 1. 'Design and Performance of Cyclone Separators,' by C. J. Stairmand, 3 p.m., Latin Theatre, The University, Edmund Street, Birmingham.

December 4. 'Design of Process Equipment with Special Hygienic Requirements,' by J. Matthews, H. F. Goodman and G. H. Botham, 5.30 p.m., Geological Society, Burlington House, Piccadilly, London, W.1.

## Society of Chemical Industry

November 20. Chemical Engineering Group. 'Recent Trends in Chemical Engineering,' by Prof. D. M. Newitt, 5.30 p.m., Burlington House, Piccadilly, London, W.1.

December 6. Chemical Engineering Group. 'The Application of Chemical Engineering to the Fertiliser Industry,' by R. P. Ross, Bristol.

## The Chemical Society

November 8. 'Synthesis of Sugars,' by Dr. J. K. N. Jones, 5.30 p.m., Chemistry Lecture Theatre, The University, Sheffield.

November 15. 'Mould Metabolic Products,' by Prof. A. Robertson, 7.30 p.m., Chemistry Department, Marischal College, Aberdeen. Joint meeting with the Royal Institute of Chemistry and the Society of Chemical Industry.

November 16. 'Transfer of Hydrogen between Organic Compounds,' by Prof. R. P. Linstead, 5.15 p.m., Chemistry Department, United College, St. Andrews.

November 16. 'Aluminium Halide as a Reagent in Organic Chemistry,' by Dr. G. Baddeley, 7.15 p.m., Royal Technical College, Glasgow.

November 22. 'Some Recent Developments in the Chemistry of Free Radical Reactions in Solution,' by Prof. D. H. Hey, 6.30 p.m., Department of Chemistry, The University, Leeds.

November 23. 'Recent Work on Inorganic Oxides,' by Dr. J. S. Anderson, 5 p.m., Washington Singer Laboratories, Prince of Wales Road, Exeter.

November 23. 'Transfer of Hydrogen between Organic Compounds,' by Prof. R. P. Linstead, 5.30 p.m., Chemistry Building, King's College, Newcastle-upon-Tyne.

November 26. 'Chemistry and Atomic Power,' by Dr. R. Spence, 8.15 p.m., Physical Chemistry Laboratory, South Parks Road, Oxford.

December 6. 'Chemistry and Atomic Power,' by Dr. R. Spence, 5.45 p.m., Department of Chemistry, University College

of North Wales, Bangor. Joint meeting with the Royal Institute of Chemistry.

December 7. 'Some Recent Developments in the Chemistry of the Heaviest Elements,' by Dr. J. S. Anderson, 5 p.m., Physics Department, University College, Southampton.

## Royal Institute of Chemistry

November 7. 'Manufacture of Glass,' by G. M. Davidson, 7 p.m., Isleworth County School, Ridgway Road, Isleworth.

November 26. 'Corrosion Processes, Their Causes and Prevention,' by Dr. F. Wormwell, 6.45 p.m., Woolwich Polytechnic, London, S.E.18.

November 29. 'Separation of Gases by Low Temperature Methods,' by Dr. J. B. Gardner, 7.30 p.m., Acton Technical College, High Street, London, W.3.

## Fertiliser Society

November 8. 'The Cement Sulphuric Acid Process,' by J. Manning, 2.30 p.m., Tudor Room, Caxton Hall, Caxton Street, London, S.W.1.

## Institution of Mechanical Engineers

November 9. 'Design of Large Pumping Installations for Low and Medium Heads,' by G. A. Wauchope and H. P. Humphreys, 5.30 p.m., The Institution, Storey's Gate, St. James's Park, London, S.W.1.

November 16. 'Some Fuel and Power Projects,' by H. Roxbee Cox, 5.30 p.m., The Institution, Storey's Gate, S.W.1.

November 23. 'Control of Boilers Fired by Solid Fuels in Suspension,' by L. Young, 5.30 p.m., The Institution, Storey's Gate, S.W.1.

## Iron and Steel Institute

November 21-22. Autumn General Meeting. Sixteen papers on iron and steel. Institute Offices, 4 Grosvenor Gardens, London, S.W.1.

## Institute of Petroleum

November 14. 'Detergents from Petroleum,' by S. F. Birch, 5.30 p.m., Manson House, 26 Portland Place, London, W.1.

November 20. 'The Petroleum Chemical Industry of N.-W. England,' by Dr. H. K. Whalley, 6.30 p.m., Engineers' Club, Albert Square, Manchester.

November 29. 'Methods for Expressing the Viscosity-Temperature Relationships of Lubricating Oils,' by F. T. Blott and C. G. Verver, 5.30 p.m., Manson House, Portland Place, London, W.1.

## Liverpool Engineering Society

November 14. 'Water Treatment for High Pressure Boilers,' by E. L. Streatfield, 6 p.m., Society's rooms, 24 Dale Street, Liverpool.

November 28. 'Manufacture and Application of Mineral Wool,' by A. M. MacGregor, 6 p.m., Society's rooms, 24 Dale Street, Liverpool.

## Oil and Colour Chemists' Association

November 9. 'Applications of Epoxide Resins in Surface Coatings,' by Dr. R. C. Aickin, 6.30 p.m., Liverpool Engineering Society, 24 Dale Street, Liverpool.

December 3. 'Some Peculiar Properties of Industrial Dusts,' by Prof. H. V. A. Briscoe, 7 p.m., Royal Station Hotel, Hull. Joint meeting with the Royal Institute of Chemistry.

## Institute of Metals

November 6. 'Pressing and Sintering of Metal Powders,' by J. C. Chaston, 6.30 p.m., Metallurgy Department, University College, Singleton Park, Swansea.

November 8. 'Transformations in Metals,' by Prof. C. S. Barrett, 7 p.m., Royal School of Mines, Prince Consort Road, London, S.W.7.

November 12. 'Metals for Gas Turbines,' by J. M. Robertson, 6.30 p.m., Institution of Engineers and Shipbuilders in Scotland, 39 Elmbank Crescent, Glasgow, C.2.

November 22. 'Metal Economics,' by Prof. A. J. Murphy, 7 p.m., James Watt Memorial Institute, Great Charles Street, Birmingham.

December 4. 'Solidification of Castings,' by R. W. Ruddle, 6.30 p.m., Metallurgy Department, University College, Singleton Park, Swansea.

## Leeds Metallurgical Society

December 6. Films. 'Process Heating Equipment,' 7 p.m., Chemistry Department, The University, Leeds.

## Liverpool Metallurgical Society

November 13. 'Welding Metallurgy,' by Prof. E. C. Rollason, 7 p.m., Lecture Theatre, Electricity Service Centre, Whitechapel, Liverpool.

## Institute of Fuel

November 12. 'Survey of Riveted and other Pressured Vessels by the Supersonic Method,' by W. W. Campbell and R. H. Murfitt, 6.30 p.m., King's College, Newcastle.

November 12. 'Fuel Oil in Industry,' by T. O. Ambrose, 7.30 p.m., The University, Edmund Street, Birmingham.

November 20. 'Automatic Control Systems for the Coal Feed of Gas Producers,' by Dr. S. A. Burke and G. A. Sparham, 5.30 p.m., Institution of Mechanical Engineers, Storey's Gate, St. James's Park, London, S.W.1.

November 21. 'Comparative Tests on Commercial CO<sub>2</sub> Recorders,' by L. J. Flaws and W. Hill, 6.30 p.m., Engineers' Club, Manchester.

December 6. 'The Upgrading of Minerals with Special Reference to Coal,' by E. M. Myers, 7.15 p.m., Gas Showrooms, Nottingham. Joint meeting with the Society of Chemical Industry.

## Hull Chemical and Engineering Society

December 4. 'Carbon Black,' by H. J. Collyer, 7.30 p.m., Church Institute, Hull.

**Manchester Literary and Philosophical Society**

November 19. 'Industrial Applications of Atomic Energy and its Products,' by Sir John Cockcroft, 5.30 p.m., The Great Hall, College of Technology, Manchester.

**Manchester Metallurgical Society**

November 21. 'Spectrographic Analysis,' by W. Ramsden, British Non-Ferrous Metals Research Association, 6.30 p.m., Engineers' Club, Albert Square, Manchester.

**Manchester Statistical Society**

November 16. 'Statistical Methods in the Design of Specifications,' by R. F. H. Bannister, 6.45 p.m., Albert Hall, Peter Street, Manchester.

**Manchester Association of Engineers**

November 16. 'Engineering Inspection and Metrology,' by W. E. Landon, 6.45 p.m., Engineers' Club, Albert Square, Manchester.

November 21. 'Sterile Filling and Filtering as applied to the Brewery Trade,' by G. Osgood, 7 p.m., Engineers' Club, Albert Square, Manchester.

December 5. 'Grinding and Grinding Machines,' by G. H. Asbridge, 7 p.m., Engineers' Club, Albert Square, Manchester.

**Plastics Institute**

November 30. 'Survey of Polystyrene Developments,' by N. G. H. Thomas, Monsanto Chemicals Ltd., 6.45 p.m., Engineers' Club, Albert Square, Manchester.

**Incorporated Plant Engineers**

November 6. 'Oil Firing,' by an engineer from Shell-Mex and B.P. Ltd., 7 p.m., Royal Society of Arts, John Adam Street, Adelphi, London, W.C.2.

November 6. 'Manufacture of Glass and its Applications,' by representative of Pilkington Bros. Ltd., 7.15 p.m., Institute of Engineers, Park Place, Cardiff.

November 7. 'Efficiency of Combined Heat and Power,' by A. C. Dunningham, 7.30 p.m., Polygon Hotel, Southampton.

Organisations convening meetings of interest to chemical engineers and chemists are invited to send full details at least two months in advance to the Editor, **INTERNATIONAL CHEMICAL ENGINEERING**, 17 Stratford Place, London, W.1. No charge is made for publication of such details.

November 8. 'The Plant Engineer and the Factories Act,' by F. W. Thompson, 7.30 p.m., Roadway House, Oxford Street, Newcastle-upon-Tyne.

November 13. 'Metallising in relation to Plant Engineering,' by L. Manuel, Metallizing Equipment Co. Ltd., 7.15 p.m., Engineers' Club, Albert Square, Manchester.

November 14. 'Combined Heat and Power Installations,' by W. O. Goldthorpe, 7 p.m., Welbeck Hotel, Nottingham.

November 15. 'Industrial Ventilation,' by L. Gordon Davies, 7.15 p.m., Radiant House, Bold Street, Liverpool.

November 21. 'Corrosion Problems,' by Dr. S. G. Gyngell, 7.15 p.m., Grand Hotel, Bristol.

November 26. 'Fuels and Firing Appliances,' by Dr. E. G. Ritchie, 7.30 p.m., The University, Leeds.

November 29. 'Mechanical Handling,' by D. Roebuck, 7.30 p.m., Grand Hotel, Sheffield.

November 30. 'Hydraulics and the Plant Engineer,' by F. H. Towler, 7.30 p.m., Imperial Hotel, Birmingham.

November 30. Annual Dinner. 'Education for Industry, the Missing Link,' by Dr. J. Bronowski, Grand Hotel, Bristol.

**Institution of Works Managers**

November 13. 'Some Impressions relative to Works Management obtained as Leader of the British Non-Ferrous Metals Anglo-American Productivity Council Team,' by W. F. Brazener, 7 p.m., Grand Hotel, Birmingham.

December 6. 'Work Study,' by W. K. Hall, I.C.I. Ltd., 7.30 p.m., Vane Arms Hotel, Stockton.

**INTERNATIONAL CONFERENCES**

November 25-30. Annual Meeting of the American Society of Mechanical Engineers, Atlantic City, New Jersey, U.S.A.

December 2-5. Annual Meeting of the American Institute of Chemical Engineers, Atlantic City, New Jersey.

**Nitric replaces sulphuric in phosphate manufacture**

That serious attention is being given in the U.S. to the conservation of sulphuric acid in fertiliser manufacture is shown by a recent article on the use of nitric acid as a substitute for sulphuric in the manufacture of phosphates. Written by J. W. Turrentine of the American Potash Institute, it appears in *Chemical and Engineering News* (1951, 29 (34), pp. 3454-56). Turrentine argues that the substitution of sulphuric by nitric is fully justified by the basic economy which it represents, namely the elimination of acid costs through the purchase of anhydrous ammonia and its re-sale as nitrates.

Considered from the viewpoint of phosphate production, the procedures he outlines place emphasis on the *maximum* production with the *minimum* of nitrogen consumption. Thus, instead of the free acidity of the chemical system being neutralised, some 40% of the nitric acid employed is evolved for recycling.

To obtain in one step the efficient absorption of nitrogen gases to nitric acid and the utilisation of this acid as formed in dissolving phosphate rock, it is recommended that the absorption tower be filled with pebble phosphate and that the gases be passed upward through the bed counter-current to a downward flow of water. Thus a solution is formed having a maximum concentration in nitric and phosphoric acid obtainable in the presence of phosphate rock, with the minimum excess of water subsequently to be evaporated. This phosphate rock, representing some two-thirds of the total, does not have to be dried and represents a further economy in that it does not have to be ground.

The tower solution as obtained is still an active absorbent of nitrogen gases, permitting a further build-up in nitric acid. After being so fortified, it is applied to ground phosphate rock to produce a slurry of monocalcium phosphate and calcium nitrate.

This slurry, on heating to 150°C., is converted to a mixture of dicalcium phosphate and calcium nitrate with the evolution of free water and some 40% of its nitric acid equivalent to yield a product, calcium nitrophosphate, containing approximately 7% N and 28% P<sub>2</sub>O<sub>5</sub>.

To reduce the cost of equipment, the suggestion is made that ceramics be substituted for stainless steel, at least on the pilot-plant scale.

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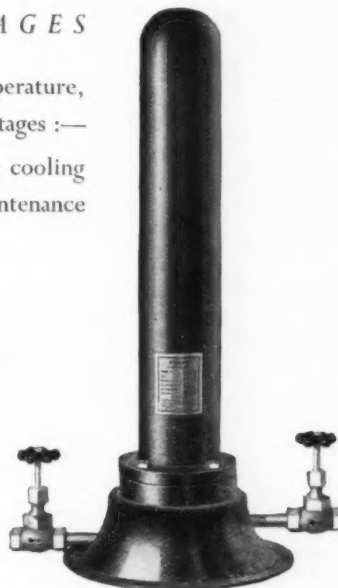


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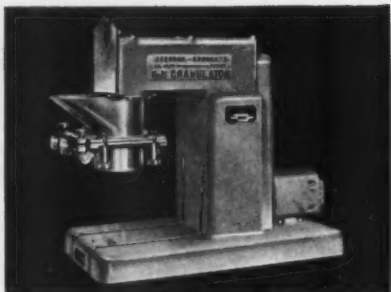
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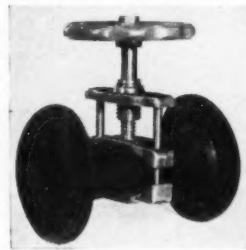


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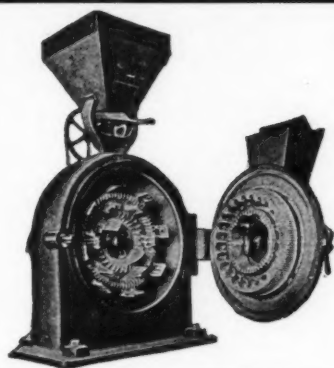
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